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The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer. Part II: User's Guide

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THE STANDARD CALIBRATION INSTRUMENT AUTOMATION SYSTEM FOR THE ATOMIC ABSORPTION SPECTROPHOTOMETER PART II - USER'S GUIDE

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This publication contains the user's guide to the EPA Atomic Absorption Instrument Automation System. This system can receive lists of samples from the Sample File Control System and returnthe measured concentrations to that system. It is capable of interfacing with a number of commercially available atomic absorption instruments. It incorporates the following major features: 1) multi-point calibration using first, second, or third degree regression or linear interpolation, 2) timely quality control assessments for spiked samples, duplicates, laboratory control standards, reagent blanks and instrument check standards, 3) reagent blank subtraction, and 4) poltting of calibration curves and raw data peaks.

The programs of this system are written in Data General Extended BASIC, Revision 4.3, as enhanced for multi-user, real-time data acquisition. They run in a Data General Nova 840 minicomputer under the operating system RDOS, Revision 6.2. This document contains detailed descriptions and examples of the features of the system, as they appear to the user.

Two related publications are "The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer Part I - Functional Specifications and "The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer Part III - Program Documentation."

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory - Cincinnati, conducts research to:

- Develop and evaluate techniques to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.

- Investigate methods for the concentration, recovery, and identification of viruses, bacteria and other microbiological organisms in water; and to determine the responses of aquatic organisms to water quality.

- Develop and operate an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.

- Develop and operate a computerized system for instrument automation leading to improved data collection, analysis, and quality control.

This report was developed in the Advanced Instrumentation Section of the Environmental Monitoring and Support Laboratory (EMSL) in the interest of advancing laboratory techniques and quality control through computerization.

Robert L. Booth
Acting Director
Environmental Monitoring and
Support Laboratory - Cincinnati

ABSTRACT

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SECTION 1

INTRODUCTION

This is a user's guide for the atomic absorption automated instrument system utilizing the standard calibration analysis method. The particular system described in this user's manual will be referred to as the ISC system which stands for Instrument Standard Calibration System. The automation system may, therefore, be adapted to any instrument system utilizing a standard calibration method of computation.

The ISC system is used to (1) develop a sample wheel pattern composed of standards, unknowns, and quality control samples, (2) analyze or measure the samples to determine sample concentrations and QC acceptability, and (3) report the final results of the analysis in the appropriate manner.

The ISC system is organized as a three level command structured system. Level 1 is the Automation System Command Level (ASCL); level 2 is the Instrument Run Command Level (IRCL); and level 3 is the Finish Command Level (FCL). At each level, various options will be available to the analyst. The purpose of this manual is to explain the overall functions of each command level and to describe the interactive sessions invoked for each and every selectable option.

The manual is designed so that the user can obtain a general overview of the structure of the system, obtain a more specific knowledge of the major command functions and reference a detailed description of each system sub-function.

Table 1 is provided as a preview of all the primary and some of the secondary command options and their purposes. Figure 1 will serve as a graphic reference to the overall structure of the system. The abbreviations noted in Table 1 are very important since they will be used repeatedly throughout the manual to locate major command levels as well as subcommand or option levels in the system. The references and appendices supplied at the end of this manual may be useful in refamiliarizing the user with the standard calibration determination method as well as the quality control assessment techniques.

1.1 DEFINITIONS

The following definitions are included to clarify word useage in this manual. The list is brief since the majority of critical terms are sufficiently defined in the reference documents 1, 2, 3 and the attached appendices.

Definitions of some terms used in this manual:

- Analog-interfaced instrument: an analytical measuring device whose output information is a continuous voltage representing sample absorbance or transmittance.
- Automatic Sequence: a complete pass through a sample wheel pattern with a constant number of replicate measurements taken at each wheel position.
- Calibration: the mathematical process by which one or more standard sample measurements are used to create an equation which relates absorbance to concentration.
- Determination: the concentration result obtained from the analysis of an unknown sample.
- Intelligent instrument: a micro or minicomputer based analytical measuring device capable of collecting and reducing analog signals to single-number absorbance values. Usually the instrument will output the information in an ASCII format.
- Measurement: the process by which an instrument response (gathered during sample atomization) is collected and reduced to a single number absorbance value. This value is also called the sample measurement.
- Replicate: a single measurement of a sample in a wheel pattern. One or more measurements may be made for a given sample and these measurements will be called replicate measurements.
- RUN: the measurement of a group of samples formed into a wheel pattern and the determination of sample concentrations through the standard calibration computational method.
- Wheel pattern: a well defined sequence of standard, unknown and quality control samples.
- Wheel position: the numerical location of a sample in a predefined sample wheel pattern.

1.2 GENERAL PRINCIPLES OF THE SYSTEM

There are several general principles which govern the ways in which the analyst may respond to computer prompts. These include procedures for inputting data, answering questions, using default conventions, asking the computer for help, and exiting an interactive session. This section will describe the general nature of these principles while other sections in this manual will be dedicated to more specific procedural descriptions.

The analyst will communicate with the computer via a user terminal. All user-responses must be followed with a RETURN. Two types of mistakes are possible when the analyst responds to computer prompts. One occurs when the analyst types a command or inputs data which the computer determines to be erroneous. In such cases the computer will generally "beep" and reprompt the analyst. The other type of mistake occurs when the analyst incorrectly inputs a parameter or data item and the computer assumes proper entry of the item. The computer will normally ask for parameter or data items in groups and after these items are input by the analyst, it will ask if any mistakes have been made. If the analyst has made an error, he my repeat the input session and correct the original mistake.

An input error can be corrected if the analyst notices it before pressing the RETURN. If the analyst presses the RUBOUT or DELETE key, the last character typed will be erased. Typing a backslash (\) will eliminate the entire input line and the analyst may reenter the answer.

The majority of computer prompts for this system are command or option lines. Therefore, the analyst may select one of many system functions from a given command or option line. The HELP or 'H' command will always be available in these situations. If the analyst inputs an 'H' at the terminal, the computer will respond with a list of options and an explanation of what each option represents. After the list is generated to the terminal, the computer will reprompt the analyst and the analyst will be required to specify one of the options.

Most computer prompts will have "default" capabilities. This means that if the analyst presses the RETURN without keying in any answer, the computer will assume an answer for the prompt. The most prevalent use of the default occurs at command or option lines where help ([H]) is the assumed default. Square brackets ([#]) are always used to denote the default parameter or option. The analyst should be aware of the fact that help is not always invoked by pressing the RETURN so the prompt should be read before a response is made.

The analyst may note that the pound sign '#' is used regularly in the system to indicate that an additional command will be required of the analyst who chooses the marked option. For example 'U#' indicates that if the analyst selects option 'U', he will be expected to select the suboption '#' which may be a 'K', a 'D', or an 'O'. (See Section 5.6.) The system will allow for "cascaded" commands so that by typing 'UK' at the major option line, the analyst can circumvent the need to respond to a suboption prompt. In a few cases as many as three questions may be answered from the primary command or option line with a cascaded response from the analyst.

The ESCape key is a very special button on the user terminal. It is the only means by which the analyst can interrupt the computer in the middle of a task. Normally the analyst who presses the ESCape key has encountered a problem in a RUN or wishes to cancel a previous request. Normally, pressing the ESCape key will immediately shift control to a major command line where the computer will wait for instructions. In a few cases, the computer will finish a task before responding to the interrupt. (This feature has been included to protect data and maintain continuity during movement from one program to another.)

Pressing the ESCape will exit the current task but will not exit the system or any of its major levels. The analyst will be required to key in a command to exit a command level. A series of exit commands will ultimately result in an exit from the system. In this case the system will automatically log off and the analyst will be required to follow the log-on procedure described in Section 2 in order to reenter the system.

At any time during the operation of this system, the computer will be performing thousands of operations for several users. Consequently, slight

delays may be noticed by the analyst in the execution of particular system functions. These delays are normal and the analyst must exercise patience before giving up and pressing the ESCape. Delays greater than 2 minutes should be made known to the system manager.

Finally, the analyst will note that blank pages have been included at the end of this manual for the purpose of recording errors, suggestions, and beneficial techniques detected while using the system. If an error occurs while using the system, it should be recorded and the analyst should immediately notify the system manager. If the problem relates to form or procedure, the analyst should describe the desired enhancement. These comments will be collected after the system has been operational for a period of time and a revision will be performed on the system to upgrade its capabilities. In addition, the analyst may find through experimenting with the system that beneficial techniques, not described in this manual, may be performed which speed up the analysis or improve data quality. These techniques may be noted so that future system user's may benefit from these experiments.

TABLE 1

SYSTEM COMMANDS AND OPTIONS

ASCL Level 1 - Automation System Command Level

T - Temporary Run File Developer (Non-SFC)

(N)S - N standards
(N)U - N unknowns
F(P) - Spike of unknown in position P
C(P) - Check of standard in position P
D(P) - Duplicate of unknown in position P
L - Lab control standard
R - Reagent blank
B - Instrument blank
X - Finish wheel pattern builder successfully
A - Abort/restart pattern builder
Q - Quit/give-up building pattern

I - Instrument checkout

R - Report/data dumper
D - Dump system data on last RUN
E - Output last Run File error list
W - Output last wheel pattern
C - Output last calibration report
R - Output last interim report
P - Output last peak plot list
F - Output last Final Report
O - Output specified Run File or Run Result File

P - Plot options
C - Calibration curve plotting
Z - Forced-zero linear curve
L - Regular linear
Q - Quadratic
C - Cubic
I - Interpolate
P - Peak plotting

S - Start procedures for making a RUN

2 - Enter Instrument Run Command Level (2) at previous status

TABLE 1 (Contd.)

X - Exit ASCL

IRCL Level 2 - Instrument Run Command Level

M(POS) - Measure absorbance for sample in position POS
C - Calibrate standards and produce report
R - Generate interim RUN status report
E# - Edit absorbance or dilution data
S(POS) - selective absorbance deletion
P - absorbance data purge for selected samples
D(POS) - modification of dilution volumes for selected sample
3 - Access Level 3 - FCL for report generation
A - Restart automatic RUN at selected position
U#- User modification of RUN-modes
K# - Modify computation mode
Z - Force zero linear
L - Regular linear
Q - Quadratic
C - Cubic
I - Interpolate
D - Modify data acquisition mode
O# - Modify output mode
T - Terminal only
L - Lineprinter only
B - Both lineprinter and terminal
N - Neither lineprinter or terminal
P - Plotting options C or P (when applicable)
1 - Exit IRCL (2) to ASCL (1)

FCL Level 3 - Finish Command Level

L - Modify BDL/GTL limits for RUN
B - Specify reagent blank absorbance levels for selected samples
G - Generate Run Results file for RUN (SFC)
F - Generate Final report for RUN (non-SFC)
2 - Exit from FCL (3) to IRCL (2)

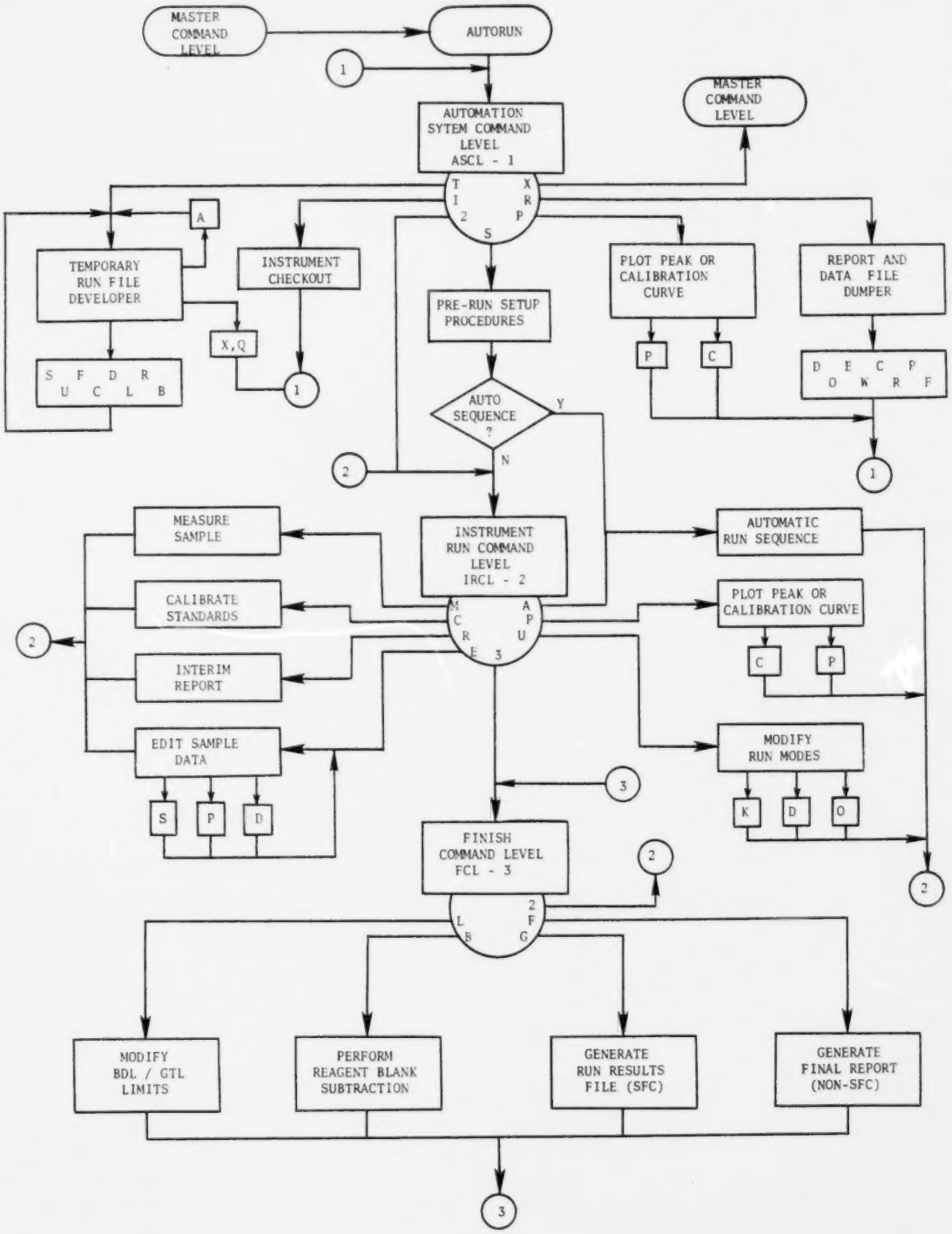


Figure 1. System usage chart.

SECTION 2

ENTERING THE SYSTEM

Most users of the ISC system will have an automated instrument, an interface box linking the instrument to the lab automation computer, and a dedicated computer terminal linking the user to the lab automation computer. To start the ISC system, three tasks must be performed: (a) the instrument must be turned on and "warmed up;" (b) the interface box must be turned on; and (c) the user must "log on" to the lab automation computer. This manual presumes that steps a and b have been performed and will describe the step c log on procedure.

In order to log on to the computer, the analyst must turn on his computer terminal. When the terminal is warmed up, the user presses the ESCape key at the upper left of the keyboard. The computer will acknowledge the escape by printing the words "ACCOUNT ID:" on the terminal. The analyst must then type in his ISC system user code. The computer will not display this code word since it is priviledged. When the user has typed the code, he will press the RETURN key on the right side of the keyboard. If the account identifier is not valid the computer will generate an error message and the user should try the log on procedure again. The system manager should be contacted if the user is unable to enter the system.

If log on is successful, the computer will display the date and time and may generate a standard message which is updated regularly by the system manager.

If the user is involved with Sample File Control analyses, he will be given a list of SFC Master Command Level (SFCMCL) options to choose from. The SFCMCL functions are described in detail in another user's guide.[4] One of the SFCMCL options is called AUTORUN. The ISC system, itself, may be accessed by selecting the AUTORUN option of SFCMCL or it may be directly accessed in those systems not affiliated with SFC. The remainder of this document will be dedicated to explaining the functions of the AUTORUN option.

BEGIN FIRST LINE OF TEXT HERE
TYPING GUIDE SHEET
CENTER OF PAGE
TOP OF IMAGE AREA
DROPPED HEAD. BEGIN SECTIONS HERE

SECTION 3

AUTOMATION SYSTEM COMMAND LEVEL

To signify entry to the ISC system, the computer will display a welcoming message to the user's terminal. It will then display the Automation System Command Line and await a user response. By typing an "H" or pressing the RETURN, the user can get the computer to show more information (see Figure 2) on the possible options available at the ASCL. Sections 3.1 through 3.5 will describe the T, I, P, R, and 2 options while Section 4 will be dedicated to describing option S.

If the user enters an 'X' at the ASCL, the computer will either sign off (non-SFC applications), or the computer will transfer control to the SFCMCL options. In addition, if no response is made by the user at this level within 30 minutes, the computer will automatically force an exit from the automation system in order to allow other users to have access to the system.

3.1 TEMPORARY (NON-SFC) RUN DEVELOPER - COMMAND 'T'

A special Run File developer option is available to analysts operating out of the realm of SFC. This ASCL option will facilitate the creation of a file which fully describes a sample sequence or RUN. If the 'T' option is chosen, the analyst will be required to perform the following tasks:

1) enter related parameters
2) edit quality control default limits
3) define the RUN wheel pattern.

The system will then assist the analyst in outputting the Run File before exiting to ASCL.

3.1.1 Entering Initial Data

On entering the Temporary Run File developer, the following messages are displayed to the user terminal:

```
BEGIN TEMPORARY RUN FILE DEVELOPER.
PLEASE ENTER INITIAL DATA.
```

The analyst will then be expected to except or input four string parameters representing the analyst's initials, the PARMETH[1] code for the RUN, the units used in the RUN, and the project code prefix for sample identifiers used in the RUN. An example input session is shown in Figure 3.

BOTTOM OF IMAGE AREA, OUTSIDE DIMENSION FOR TABLES AND ILLUSTRATIONS
BEGIN LAST LINE OF TEXT
3/8"

PAGE NUMBER
EPA-267 (Cln.) (4-76)

```
**************************
*        GSC0            *
* STANDARD CALIBRATION   *
* INSTRUMENT AUTOMATION  *
*       SYSTEM           *
**************************

AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > H
   S  - START INSTRUMENT AUTOMATION RUN SEQUENCE
   I  - INSTRUMENT CHECKOUT
   P# - PLOT CALIBRATION OR PEAK DATA (C OR P)
   R  - DUMP REPORT FILE (SELECTABLE)
   X  - EXIT AUTOMATION SYSTEM (TO MASTER COMMAND LEVEL)
   T  - TEMPORARY RUN FILE CREATOR (NON-SFC)
   2  - ACCESS IRCL(2) AT PREVIOUS RUN STATUS

AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) >
```

Figure 2. Automation system command level options.

```
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > I

THE TEMPORARY RUN FILE CREATOR IS AT YOUR SERVICE!

PLEASE ENTER INITIAL DATA!

YOUR INITIALS (3) - [BJM] > DPR
PARMETH CODE (7)  - [229YZ02] > 224YZ02
UNITS (UG OR MG/L)- [UG/L] >
PROJECT CODE (4)  - [EMSL] >

MISTAKES ON PRELIMINARY DATA ? ([N] OR Y) >

DEFAULT-TO-OLD OR ENTER-NEW QC LIMITS ? ([D] OR E) >

PREPARE TO BUILD YOUR WHEEL PATTERN!

GENERATE OR ENTER ID'S ? ([G] OR E) > G

SEED NUMBER FOR UNKNOWN IDS. > 134

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) >
```

Figure 3. Preliminary inputs for the temporary Run File development session.

The character length of each parameter is specified exactly. Errors in length will cause the computer to reprompt for the parameters.

3.1.2 Editing Quality Control Limits

The temporary Run File developer can be used to edit a set of quality control limits for spikes, duplicates, reagent blanks, and control standards. The last used limits may be accepted or a complete edit may be performed on the existing limits. Figure 4 shows an example session in which the current QC limits are edited.

3.1.3 Building a Wheel Pattern for a RUN

The next step in developing a Run File will be to build a wheel pattern. It will be recommended that the analyst have a written list of sample types and corresponding wheel positions available before starting this session. This is especially necessary when duplicates, spikes and/or check standards are to be measured during the RUN. The wheel pattern builder begins with the message:

```
PREPARE TO BUILD YOUR WHEEL PATTERN
GENERATE OR ENTER IDS? ([G] or E)  >
```

If the analyst elects to have the sample numbers generated automatically, he will be required to input a seed number for the first unknown. Unknown samples will then be assigned sample numbers starting with PROJ0376 and incrementing by 1 for each unknown specified in the wheel pattern. If the analyst elects to enter the unknown identifiers, he will be responsible for typing in a one to four digit number for each unknown in the wheel pattern. The computer will "pad" the number so that all sample identifiers will have 8 characters of the form PROJ0002 or PROJ7653.

In order to build a wheel pattern, the analyst will be required to specify which type of sample will occupy which position in the wheel. There will be a few restrictions placed on the number of wheel positions which may be filled or the type of sample which can occupy a particular wheel position. Figure 5 shows the list of Run File development options available to an analyst building a wheel pattern. One of eight sample types (S, U, F, C, D, L, R, or B) must be selected to fill each particular wheel position.

For example, by typing an 'S' on the terminal at the start of a session, the analyst specifies that a standard sample is to occupy wheel position number 1. The computer will keep track of each specified sample and show the wheel position of each sample as the pattern is built. The computer will assist in processing each selected option and will always return to the Run File developer option line and wait for another command.

A special feature has been devised for standard and unknown samples so that a group of samples may be entered with one command. If a number (N) is typed before the letter 'S' or 'U', the computer will automatically assign the next N standards or unknowns to the available wheel positions. Figures 6 and 7 show the uses of these commands. In addition to the command, the

```
DEFAULT-TO-OLD OR ENTER-NEW QC LIMITS ? ([D] OR E) > E ↵

FOR LABORATORY SPIKES:

UCL - [ 122.5 ] > ↵
UWL - [ 115 ] > ↵
MEAN- [ 100 ] > ↵
LWL - [ 85 ] > ↵
LCL - [ 77.5 ] > ↵
MISTAKES ON SPIKE LIMITS ? ([N] OR Y) > ↵

FOR LABORATORY REAGENT BLANKS:

UCL - [ 2 ] > 3 ↵
UWL - [ 1.5 ] > 2 ↵
MEAN- [ .5 ] > 0 ↵
LWL - [-.5 ] > -2 ↵
LCL - [-1 ] > -3 ↵
MISTAKES ON REAGENT BLANK LIMITS ? ([N] OR Y) > ↵

FOR LABORATORY DUPLICATES:

SLOPE     - [ 1.5E-02 ] > .02 ↵
INTERCEPT- [ .5 ] > 2 ↵

R = XBAR * 2E-02 + 2

MISTAKES ON DUPLICATE PARAMETERS ? ([N] OR Y) > ↵

FOR LABORATORY CONTROL STANDARDS:

UCL - [ 115 ] > ↵
UWL - [ 110 ] > ↵
MEAN- [ 100 ] > ↵
LWL - [ 90 ] > ↵
LCL - [ 85 ] > ↵
MISTAKES ON CONTROL STANDARD LIMITS ? ([N] OR Y) > ↵
```

Figure 4. Quality Control limit edit session.

```
PREPARE TO BUILD YOUR WHEEL PATTERN!

GENERATE OR ENTER ID'S ? ([G] OR E) > G ↵

SEED NUMBER FOR UNKNOWN IDS. > 134 ↵

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > H ↵
  [N]S    - CALIBRATION STANDARDS (EG. 5S)
  [N]U    - UNKNOWN SAMPLES (EG. 10U)
     F[P] - SPIKED UNKNOWN (EG. F9)
     C[P] - CHECK STANDARD (EG. C5)
     D[P] - DUPLICATE UNKNOWN (EG. D8)
     L    - CONTROL STANDARD
     R    - REAGENT BLANK
     B    - INSTRUMENT BASELINE BLANK
     X    - EXIT SESSION SUCCESSFULLY
     A    - ABORT/RESTART PATTERN BUILDER
     Q    - QUIT (#$%&+*) GIVE-UP

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) >
```

Figure 5. Temporary Run File developer wheel pattern options.

computer will require the analyst to enter concentrations for each standard sample. If sample identifiers are being manually input, the computer will require the analyst to type identifier numbers for each specified unknown sample.

Spiked unknowns, check standards, and duplicate unknowns may be included in the wheel pattern only if they reference a valid sample which occupies a previous wheel position (P). For example, a check standard must be specified such that it "points" to a calibration standard with the same concentration. Similarly, spiked and duplicated samples must reference their original unknowns so that QC comparisons may be properly performed. Figure 8 shows the use of the F, C, and D options. Note the pointer value and check Figures 6 an 7 for corresponding wheel position matches.

Lab Control Standards are specified by the letter L and additional information must be supplied to the computer as to the theoretical concentration of the sample. Lab Reagent Blanks are specified by the letter R and instrument blanks are specified by the letter B. Figure 9 shows the use of the L, R, and B options.

The analyst will specify all the samples in the wheel pattern and input the additional information for those samples requiring it. When the pattern has been completely defined, the analyst signifies completion of the session by typing an 'X'.

If difficulties arise, the analyst can either abort and restart the pattern builder with the 'A' option or he can quit with the 'Q' option. If the 'Q' option is used the computer will return control to ASCL.

3.1.4 Run File Output Options

After successfully building a wheel pattern, the analyst may have the computer generate the Run File to an appropriate output device (i.e. terminal, line printer, both terminal and line printer, or no output). Figure 10 shows an example Run File output session. The Run File contains more information than was specified during the wheel pattern generation process because the QC limits specified in the QC-edit session are included in the output (see Figure 4).

3.1.5 Exit to ASCL

Once the Run File has been output to the appropriate device, the computer will shift control to the ASCL and await another command. Normally the analyst will want to start an automated RUN using the newly created Run File (see Section 4 below).

3.1.6 Escape Sequencing

If an ESCape is pressed any time during the Run File development process the following question will be generated by the computer:

```
DO YOU WANT TO QUIT OR START OVER? ([Q] or S) >
```

```
RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > 7S )

FOR WHEEL POS   1     STD CONC =  > 0 )
FOR WHEEL POS   2     STD CONC =  > 5 )
FOR WHEEL POS   3     STD CONC =  > 10 )
FOR WHEEL POS   4     STD CONC =  > 20 )
FOR WHEEL POS   5     STD CONC =  > 30 )
FOR WHEEL POS   6     STD CONC =  > 50 )
FOR WHEEL POS   7     STD CONC =  > 100 )

MISTAKES ON CALIBRATION STANDARD VALUES ? ([N] OR Y)    > )

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) >
```

Figure 6. Calibration standards entry session for the temporary Run File developer.

```
RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > 4U )

WHEEL POS  11     SAMPLE ID > EMSL0134
WHEEL POS  12     SAMPLE ID > EMSL0135
WHEEL POS  13     SAMPLE ID > EMSL0136
WHEEL POS  14     SAMPLE ID > EMSL0137
```

Figure 7. Unknown entry session for the temporary Run File developer.

```
RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > D13 )

WHEEL POS  15

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > F13 )

WHEEL POS  16

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > C5 )

WHEEL POS  17
```

Figure 8. Duplicate, spike, and check standard entry sessions for the temporary Run File developer.

```
RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > R )

WHEEL POS   8

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > L )

WHEEL POS   9    CTL STD CONC =  > 20 )

RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) > B )

WHEEL POS  10
```

Figure 9. Reagent blank, lab control standard, and instrument blank entry sessions for the temporary Run File developer

```
RUN FILE DEVELOPMENT OPTIONS: ([H]SUFCDLRBXAQ) ) X )

YOUR RUN FILE NAME IS 0000000.RF

RUN FILE OUTPUT OPTIONS: ([H]TLBN)  ) H )
   T - TERMINAL OUTPUT ONLY
   L - LINEPRINTER OUTPUT ONLY
   B - BOTH TERMINAL AND LINEPRINTER OUTPUT
   N - NO REPORT GENERATED

RUN FILE OUTPUT OPTIONS: ([H]TLBN)  ) I )

RUN FILE 0000000.RF     14 : 7     7 / 23 / 1981

1 81  7 23 14  7 GSC0  224YZ02 DPR
3   +0.000E+00 P   224YZ02              S
3   +5.000E+00 P   224YZ02              S
3   +1.000E+01 P   224YZ02              S
3   +2.000E+01 P   224YZ02              S
3   +3.000E+01 P   224YZ02              S
3   +5.000E+01 P   224YZ02              S
3   +1.000E+02 P   224YZ02              S
2          LRB P   224YZ02              UG/L
4 LRBL     UCL P   224YZ02 +3.000E+00 MM/DD/YY
4 LRBL     UWL P   224YZ02 +2.000E+00 MM/DD/YY
4 LRBL     AVE P   224YZ02 +0.000E+00 MM/DD/YY
4 LRBL     LWL P   224YZ02 -2.000E+00 MM/DD/YY
4 LRBL     LCL P   224YZ02 -3.000E+00 MM/DD/YY
2          LCM P   224YZ02              UG/L
2          LCT P   224YZ02 +2.000E+01 UG/L
4 LCST     UCL P   224YZ02 +1.150E+02 MM/DD/YY
4 LCST     UWL P   224YZ02 +1.100E+02 MM/DD/YY
4 LCST     AVE P   224YZ02 +1.000E+02 MM/DD/YY
4 LCST     LWL P   224YZ02 +9.000E+01 MM/DD/YY
4 LCST     LCL P   224YZ02 +8.500E+01 MM/DD/YY
3          P   224YZ02              B
2 EMSL0134     P   224YZ02              UG/L          SFCRECNO BKNUM
2 EMSL0135     P   224YZ02              UG/L          SFCRECNO BKNUM
2 EMSL0136 LDX P   224YZ02              UG/L          SFCRECNO BKNUM
4 LSPK     UCL P   224YZ02 +1.225E+02 MM/DD/YY
4 LSPK     UWL P   224YZ02 +1.150E+02 MM/DD/YY
4 LSPK     AVE P   224YZ02 +1.000E+02 MM/DD/YY
4 LSPK     LWL P   224YZ02 +8.500E+01 MM/DD/YY
4 LSPK     LCL P   224YZ02 +7.750E+01 MM/DD/YY
4 LDUP     A   P   224YZ02 +0.000E+00 MM/DD/YY
4 LDUP     B   P   224YZ02 +0.000E+00 MM/DD/YY
4 LDUP     C   P   224YZ02 +2.000E-02 MM/DD/YY
4 LDUP     D   P   224YZ02 +2.000E+00 MM/DD/YY
2 EMSL0137     P   224YZ02              UG/L          SFCRECNO BKNUM
2 EMSL0136 LD2 P   224YZ02              UG/L
2 EMSL0136 LSF P   224YZ02              UG/L
2 EMSL0136 LSA P   224YZ02              UG/L
3   +3.000E+01 P   224YZ02              CS

AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) )
```

Figure 10. Run File output session for the temporary Run File developer.

In the case of the temporary Run File developer, an ESCape nullifies any previous wheel pattern specifications. If the analyst chooses to start over, the computer will restart the session with the initial data input session described in Section 3.1.1.

3.2 INSTRUMENT CHECKOUT - COMMAND 'I'

The essence of the instrument checkout function is to facilitate instrument setup procedures and to optionally perform one or more measurement cycles. The setup procedure may require establishing instrument timing parameters. The measurement cycle requires the performance of a sampling sequence and the acquisition and reduction of instrument data to obtain a single number absorbance value for the measurement.

The instrument checkout procedure is available as an option through ASCL. At this level, the module is useful for tests made by the analyst or system manager to verify the operation of interface hardware, realtime system software, and automation software. The instrument checkout procedure is also invoked just prior to an automated run (Section 4.3) in order to prepare the ISC system for subsequent measurements.

A general requirement of the instrument checkout routine is that an appropriately interfaced instrument be available and that the instrument has been "warmed up" and adjusted for the particular method and analyte being used.

Since the ISC system has been implemented on a number of commercially available instruments, each application is unique and requires its own explanation. The following subsections will describe the current applications of the ISC system.

3.2.1 Instrument Checkout with the Perkin-Elmer 5000 Flame/Flameless AA Application

The Perkin-Elmer 5000 is a very sophisticated atomic absorption instrument. It contains a microcomputer which manages the flame and flameless sample changers, the flameless time and temperature controller, the keyboard and display panel, and the data acquisition and reduction processing. Under the proper conditions the PE5000 will output the absorbance results of a sample measurement to an ASCII Character Buffer (ACB) Interface.[5] In cases such as these, the Instrument Checkout option will serve to "read" a string of output from the ACB and display the character string and the corresponding absorbance value to the user terminal.

The following conditions must be fulfilled for the ISC system to acquire and reduce output from the PE 5000: 1) the RS-232C interface in the PE 5000 must be set to single-line slow-speed transmission; 2) the ACB box must be properly connected to the PE 5000 printer output plug; 3) the PE 5000 must be set to output absorbance values; and 4) the PE 5000 PRINT button must be on. Conditions 1 and 2 will be taken care of at system installation time while conditions 3 and 4 must be activated by the analyst prior to every RUN.

The data acquisition software for this module is trivial, however, there are some points worth noting. The module will normally read one line of PE 5000 output from the ACB box unless the last two characters in the line are AZ, AV, or SD. In these cases the module will ignore the line and read the next available line. The AV (average) and SD (standard deviation) will appear in situations where replicate injections are made on a given sample in the wheel. An AZ will appear when the instrument performs an auto zero to establish a baseline. The analyst may also perform a manual auto zero by pressing the AZ button on the PE 5000 console. The line will be ignored by the checkout module in either case.

Some qualifiers at the end of a line will cause a data error message to be output to the user terminal; these include S1, S2, S3, RS, CV, ER, and C. An ER indicates that the PE 5000 has encountered a problem it cannot resolve. The rest of the qualifiers are associated with the internal standard calibration capabilities in the PE 5000 itself. The ISC system will only operate on absorbance output generated by the PE 5000.

The analyst should also note the slight difference between the flame and furnace applications on the PE 5000. The auto zero function operates differently for each application. In the flame system, the auto zero function produces a one line result with the AZ qualifier at the end of the line. In the furnace application, an actual atomization is made and a reading is output. The auto zero is then performed internally and a zero result is output with the AZ qualifier at the end of the line. This implies that all furnace runs on the PE5000 which execute a controlled auto zero at the beginning of the analysis must have an instrument blank in the first wheel position so that the ISC system can place the measurement in an inocuous position.

The instrument checkout option for the PE 5000 can be used to access characters already stored in the ACB buffer box or the option can be invoked prior to atomization or aspiration and the computer will wait for the transmission of a line of characters by the instrument. The only user action required in this application is to respond to the prompt,

DO YOU WANT TO MAKE A TEST INJECTION ([N]OR Y)>

It is not necessary to actually measure a sample for this module to be used. The READ button may be pressed on the instrument control keyboard and the contents of the display panel will be output to the ACB.

Normal exit of this routine is made when no more test injections are requested. Pressing the ESCape key at any time during the execution of this module will force an immediate transfer of control the ASCL.

3.2.2 Instrument Checkout with the Varian AA 775 Flameless AA Application

The Varian AA 775 instrument operates similarly to the PE 5000 (Section 3.2.1 above). It is equipped with a microprocessor which controls the run sequence and collects and reduces the raw peak atomization data. It outputs

a line of results through an RS232 interface and can be configured to send the results to the ACB box.[5]

The format of a line output generated by the AA 775 is different from that generated by the PE 5000. The AA 775 prefixes each output line with a device number of 65. One of four qualifiers may immediately follow this device code. They include ZERO, MEAN, SDEV and NUM. If any of these qualifiers is present the computer will disregard the entire line and prepare to read the next line. A ZERO indicates an auto zero function, while MEAN, SDEV and NUM are associated with the statistical assessment of a number of replicate measurements. One terminating character (C) will cause the computer to output an error message to the user terminal. The C indicates that the AA 775 is set in the concentration mode. As in the PE 5000 application, the instrument must be set to output absorbance values in order for the ISC system to operate on the output. A redundant check is made on the output line to certify that it is producing absorbance measurements. The tenth character in the line must be either an H or an A indicating a peak height or peak area absorbance. If an error condition occurs on the AA 775 no output is generated and the instrument stops whatever it is doing.

The prompt for the AA 775 checkout session is identical to that for the PE 5000. Similarly, simulated output can be generated by pressing the READ button. The user may terminate the checkout session by responding with an N to the test injection prompt. If an ESCape is pressed at any time, the control will revert to the ASCL.

3.2.3 Instrument Checkout with the Perkin-Elmer 403/503 Flameless AA Application

The Perkin-Elmer 403 and 503 are double beam flame and flameless atomic absorption instruments. This discussion will center on the flameless application using the HGA 2000 furnace controller and the AS-1 sample changer. Unlike the PE 5000 and Varian AA 775, mentioned in Sections 3.2.1 and 3.2.2, the PE 403/503 series instrument produces an analog signal which is transmitted to the computer and converted to a series of digitized numbers. This array of readings represent the peak atomization data accumulated during a sample burn. The computer and the instrument communicate with each other through the EPA flameless atomic abosrption interface box.

The user will be required to perform the necessary instrument set-up steps prior to utilization the instrument checkout option. In addition, the analyst must turn the AS-1 power on but leave the start button in the off position. The replicate injection counter should be set to 1.

The interface box must also be activated. This requires turning on the power and setting the MANUAL/COMPUTER switch to the COMPUTER position. The reset button should then be pressed to initialize the interface.

An example session for performing a PE 403/503 instrument checkout procedure is shown in Figure 11. On entering the option, the computer will

```
SAMPLE AND REFERENCE BEAM TEST

REFERENCE BEAM  =  8467.9
SAMPLE BEAM     =  9503.3
BEAM DIFFERENCE =  1035.4

ANOTHER BEAM DIFFERENCE TEST  ? Y )

SAMPLE AND REFERENCE BEAM TEST

REFERENCE BEAM  =  8473.9
SAMPLE BEAM     =  9509.5
BEAM DIFFERENCE =  1035.6

ANOTHER BEAM DIFFERENCE TEST  ? N )

ATOMIZATION TIME IN SECONDS  ? 8 )

DELAY TIME IS  13.3  SECONDS.
TIME BETWEEN POINTS IS  3 /60 SECONDS.
TOTAL DATA TAKING TIME IS  15  SECONDS.

DO YOU WISH TO MAKE A TEST INJECTION  ? Y )

PLACE A STANDARD IN THE CURRENT SAMPLING POSITION.

*** START SAMPLER NOW. ***


PK HT = 0.781901     PK LOC =  110.142
BSLN  = 0.740761     ST DEV =  2.1909E-04     TRUE =  4.11404E-02
```

Figure 11. Instrument checkout session for the Perkin-Elmer 403/503 Flameless Application.

immediately check the voltage levels of both analog channels and output a 10-point average of each and the difference between them. The next step requires the analyst to input the atomization time set for the run. Some latitude is allowed in specifying this time since the computer will create a data acquisition window which begins before the start of atomization and continues until after atomization is complete. After the atomization time is specified, the computer will report the time interval between readings and the total data taking time.

At this point the analyst has the opportunity to perform a test injection. In order to perform a test injection, the analyst must place a mid-range standard in the current sampling position and respond with a 'Y' to the computer prompt. The first test injection will constitute the first injection of the run and will cause the computer to generate a message to the user to start the AS-1 changer. The analyst must then press the START button on the AS-1 sampler. (The user should not need to touch the AS-1 again until the run is complete unless more than one wheel of samples is to be analyzed.) The sample will then be injected, dried, charred and atomized and a small report will be output to the user terminal detailing the results of the burn. In addition, a crude plot may be generated to the line printer or user terminal in order to verify the timing parameters. The plot session and module exit procedure are shown in Figure 12. Timing can be modified and additional test injections can be made until the user is satisified with the instrument and computer interfacing.

The analyst may exit the checkout module by answering no to the test injection prompt. If the user presses an ESCape at anytime, control will immediately revert to ASCL.

3.2.4 Instrument Checkout with the Varian AA5/AA6 and the Perkin-Elmer 403/503 Flame AA Application

The Lawrence Livermore Laboratory has developed a flame atomic absorption sample changer for the EPA which can be adapted for use on the Varian AA5/AA6 single beam instrument or the Perkin-Elmer 403/503 double beam instrument. This discussion will consider the flame application using the LLL-1 changer on either single of double beam instruments. In this application, the instrument will supply a continuous analog signal to the computer while the computer will act on user commands to control the sampling position and sampling rate. Figure 13 shows an example checkout session.

The first step in the instrument checkout routine requires that the analyst teach the computer the location of position #1 in the sample wheel. This is done by setting the sampler to the LOCAL mode and setting the arm to the up position. By using the clockwise (CW) and counter clockwise (CCW) buttons, the analyst may position the center of the arm directly over wheel position number 1. The analyst should then switch from LOCAL to COMPUTER mode. When this is done the analyst is asked to measure a blank sample to establish a preliminary baseline reading. The timing parameters must then be defined. These include the delay time between the start of sample aspiration and the sample entry into the flame. The delay time should be

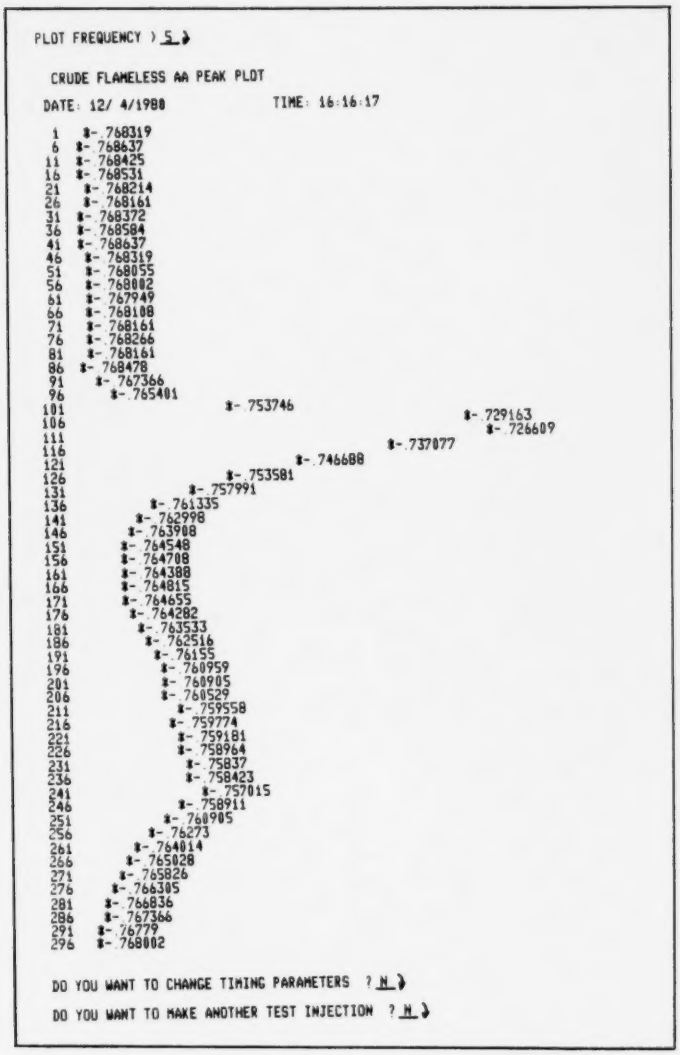


Figure 12. Generating a crude plot of a test atomization taken during an instrument checkout session.

```
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > I

SET SAMPLER ARM AT POSITION #1 AND PRESS RETURN >

PREPARE TO MEASURE BASELINE FOR FIVE (5) SECONDS.
ENTER SAMPLE WHEEL POSITION  > 1
PRESS RETURN TO BEGIN ASPIRATION  >

PRESS RETURN TO BEGIN BASELINE MEASUREMENT  >

SAMPLE BEAM    =  8192.97
BASELINE VALUE =  3.72178E-04

ANOTHER BASELINE TEST ([N] OR Y)  > N
BASELINE=  3.72178E-04

SAMPLE ASPIRATION-TO-ATOMIZATION LAG TIME IN SECONDS  > 5
SAMPLING TIME IN SECONDS  > 2

TOTAL DATA TAKING TIME IS  7  SECONDS.

DO YOU WANT TO MAKE A TEST MEASUREMENT ? ([N] OR Y)  > Y

ENTER SAMPLE WHEEL POSITION  > 2
PREPARE TO MEASURE A SAMPLE.
PRESS RETURN WHEN READY >


MEAN MEAS =  .102871          STD DEV =  4.05994E-05
BASELINE =   3.72178E-04      TRUE ABS=  .102499


DO YOU WANT TO CHANGE TIMING PARAMETERS  > N

DO YOU WANT TO MAKE ANOTHER TEST MEASUREMENT ([N] OR Y)  > N
```

Figure 13. Instrument checkout session for the Varian AA5/AA6 and Perkin Elmer 403/503 Flame Application.

sufficient to allow the output signal to equilibrate. The analyst must also specify the time necessary to obtain an average reading. This time may vary between 0.5 and 20 seconds.

Once the sampler has been justified and the timing has been specified, the analyst may test the sampling mechanism by making one or more test injections. The analyst must identify the wheel position of the sample to be measured. After the measurement has taken place, the computer will output a small report on the results of the measurement. Test injections may be repeated and timing parameters may be modified until the analyst is satisfied with the proper operation of the system.

The user may exit the checkout routine by typing N to the test injection prompt. If an ESCape is pressed during a sample arm movement, the analyst will be required to reinitialize the sampler to position #1. If ESCape is pressed at any other time, control will revert to ASCL.

3.3 PLOTTING ON APPROPRIATE GRAPHICS TERMINALS - COMMAND 'P'

The ASCL 'P' or Plot option will facilitate the display of previously generated calibration curves and raw peak array curves. The raw peak data plots will only be available on instruments which generate raw peak data during atomization. Intelligent instruments may or may not supply this type of peak-plot output.

The plot option normally requires the use of a graphics display terminal for the generation of calibration curves or raw peak data curves. One notable exception to this rule is that raw peak data may be generated in crude form if a graphics terminal is unavailable. Section 3.3.2 will describe this exception in more detail.

3.3.1 Calibration Curve Plotting ('PC')

In order to generate plots of the current calibration curves, the analyst must type a 'P' in response to the ASCL options. The computer will then ask which type of plot is to be generated. The analyst may then type a 'C' and enter the calibration curve plotting module. This sequence may be shortened somewhat by typing a 'PC' at ASCL and the calibration curve plot module will be immediately invoked.

On entering the calibration curve plot module, the analyst will be asked to define which type of calibration curve is to be generated. Figure 14 shows the available options at this level. It should be noted that some of these plots may not be available if a small number of standards were used in the calibration, for example, the calibration of two standards will have plots for the Z, L, and I options only.

Figure 15 depicts a plot of a regular linear (L) calibration curve. After the plot is generated, the analyst will be given two minutes to preview and/or copy the plot to an available hard-copy device before the screen is cleared. Pressing the RETURN key will clear the screen. After a

```
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > P
CALIBRATION OR PEAK PLOTTING ([C] OR P) >
CALIBRATION PLOTTER BEGINS
TYPE OF PLOT? ([H]ZLQCIX)  > H
   Z - FORCED ZERO LINEAR FIT
   L - LINEAR
   Q - QUADRATIC
   C - CUBIC
   I - INTERPOLATION
   X - EXIT PLOT ROUTINE
TYPE OF PLOT? ([H]ZLQCIX)  > L
```

Figure 14. Calibration curve plot session.

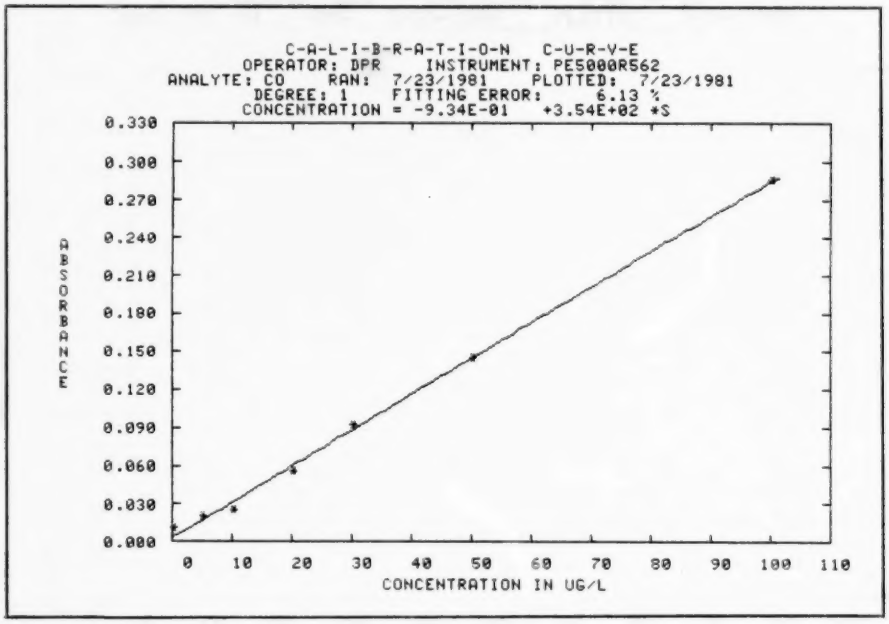


Figure 15. Standard calibration curve as generated to a graphics display device.

plot is erased another plot may be generated using a different equation. When the analyst is finished reviewing the available curves, the calibration plot routine will exit to the ASCL either through the exit command 'X' or if no respose is entered within five minutes, the routine will automatically exit to ASCL.

Pressing an ESCape at any time during the calibration curve generation process will shift control to the calibration plot options shown in Figure 14.

3.3.2 Raw Peak Array Plotting ('PP')

In order to generate raw peak-data plots, the analyst may type a 'P' in response to the ASCL options. The computer will then ask which type of plot is to be generated. The analyst may then type a 'P' and enter the appropriate module. This sequence may be shortened somewhat by typing a 'PP' at ASCL and the peak plotting module will be immediately invoked. If no peak data is available a message will be generated which indicates that the option is unavailable and control will be returned to the ASCL.

On entering the peak plotting module, the computer will ask whether the output is to be generated to a standard graphics terminal or to a line printer in crude form. In addition, the analyst will have to specify which plot is to be the first one displayed. Once these questions are answered, the computer will search for the preselected peak and display the pertinent peak parameters to the user terminal (See Figure 16). The computer will then ask if the peak is to be plotted. Figure 17 and 18 show the peak plot as generated on a graphics terminal and line printer respectively.

Once a peak plot has been generated, the computer will automatically cycle to the next available peak, output the pertinent information to the user terminal, and ask if the peak is to be plotted. The cycle will be repeated until all of the peak data has been accessed or until the analyst exits the routine.

The only way an analyst may exit the peak plotting module will be by pressing the ESCape key. In addition, the analyst may skip the output of peak parameter data by pressing the ESCape key. The computer will immediately stop printing the information and ask if the plot should be generated. An example of this feature is shown in Figure 19 along with an example of the peak plot exit function. On exiting the peak plotting module control will immediately revert to the ASCL and await further commands.

3.4 REPORT AND DATA DUMPER OPTIONS - COMMAND 'R'

At the ASCL, the user will have the opportunity to review the current files and RUN-data maintained by the system. This information will represent data and reports accumulated during the last RUN peformed by the system. This feature will normally be used by the system manager or analyst to locate problems in system variables or to examine system status. An 'O' command is available for outputting Run File or Run Results File contents to

```
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > P )
CALIBRATION OR PEAK PLOTTING ([C] OR P) > P )
TEKTRONIX OR LINEPRINTER ([T] OR L) > I )
NUMBER OF FIRST PEAK  > 19 )

                ATOMIC ABSORPTION ARRAY PARAMETERS (ISCN)

 DATE:  7/27/1981 - 10:45:43    MEASURED:  3/ 7/1980 -  9:35: 7
 ANALYST:                       ANALYTE:
 SAMPLE TYPE: SPIKED UNKNOWN    SAMPLE ID:
 PEAK NO:   19                  WHL POS:  17
 INSTRUMENT:                    MODE: FULLY-AUTO STD CAL
 METHOD: PEAK HEIGHT            BASELINE: LEADING
 A/D CHANNEL:  0                NUMB PTS IN CALL: 300
 DELAY BEFORE:  857*(1/60)      TIME BETWEEN PTS:  2*(1/60)
 NUMB ACTUAL PTS: 300
 MAX ARRAY VALUE:  -0.6561      MAX LOCATION: 126
 MIN ARRAY VALUE:  -0.8053      MIN LOCATION:  12
 AVE LEADING BSLN:  -0.8049     BSLN STD DEV:  2.509E-04
 PEAK HEIGHT:   0.0066          PEAK LOCATION: 121.61

PLOT PEAK (N[CR] OR Y)  > Y )
```

Figure 16. Peak plot output session with example peak parameter report.

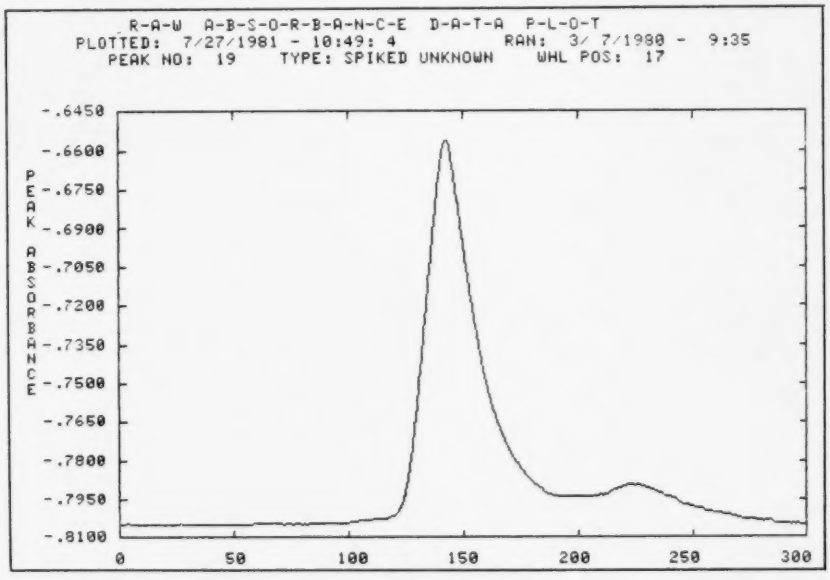


Figure 17. Peak absorbance array as generated to a graphics display device.

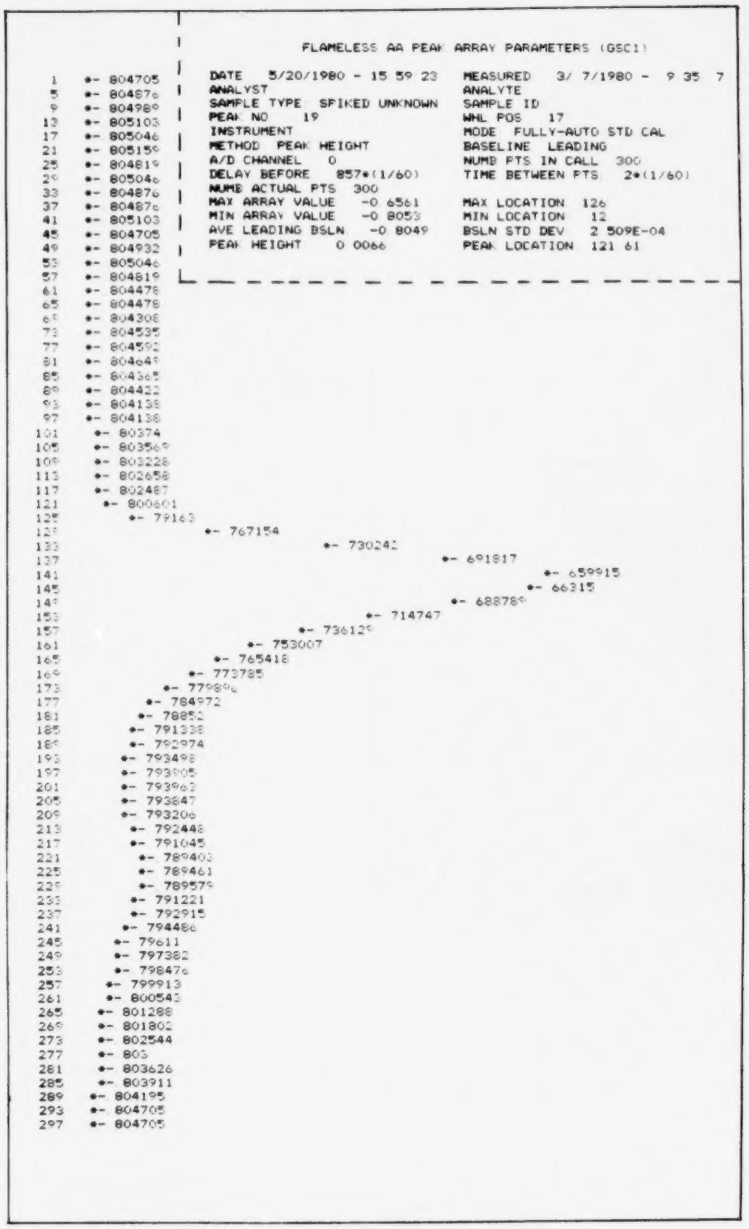


Figure 18. Peak array plot output as generated to a non-graphics device.

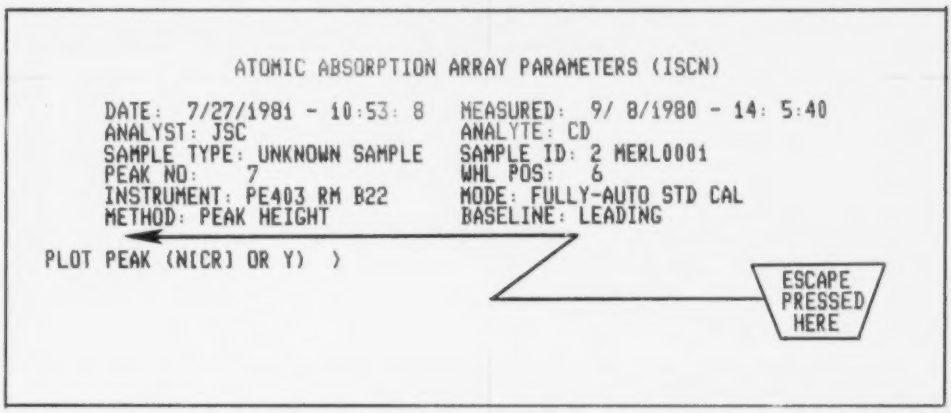


Figure 19. Escape feature in the peak plot output session.

the appropriate output device. Figure 20 shows the available report and data dumper options, Figure 21 shows an example of the 'D' option, and Figure 22 shows the usage of the 'O' option. Any "list file" may be generated using the 'O' option but it is expected the Run Files and Run Results Files will be the predominant product of the option. The full file name must be presented to the system if the file is to be successfully generated. The other reports that may be generated by this command (E, W, C, R, P, and F) will be explained in more detail later in this manual.

Pressing an ESCape at any time during the report data dumper session will return control to the main option line (Figure 21). Exiting the system is accomplished by typing a '1'. Control will then be transferred to the ASCL.

3.5 DIRECT ACCESS OF THE IRCL FROM THE ASCL - COMMAND '2'

Under special circumstances, the IRCL can be accessed directly from the ASCL. Normally, the analyst will be expected to perform all of the steps indicated in Section 4 below for processing newly devised Run Files. The analyst will be allowed to circumvent these preliminary steps and transfer control to the IRCL if, and only if, the following conditions are met:

1) the Run File has been previously translated (Section 4.1);
2) all pre-RUN parameters have been defined (Section 4.2); and
3) the Run File or the Run Results File specifying the wheel pattern has not been destroyed or altered during the interim period.

This option will allow an analyst to exit the system after a RUN and prior to final reporting without endangering the data acquired during the RUN.

If the analyst attempts to execute a '2' command from the ASCL, and the three conditions mentioned above are not met, then the computer will indicate the error and return to the ASCL command line and await another command.

```
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > R ↵

BEGIN REPORT / DATA DUMP SESSION.

REPORT/DATA OUTPUT OPTION: ([H]DWCRFEO1) > H ↵
   D - DUMP CONTENTS OF DATA FILES .DF, .ID, AND .TR
   W - OUTPUT WHEEL PATTERN
   C - OUTPUT CALIBRATION REPORT
   R - OUTPUT INTERIM REPORT
   F - OUTPUT FINAL REPORT
   E - OUTPUT RF TRANSLATOR ERROR LIST
   O - OUTPUT OTHER LIST FILES (I.E. .RF AND .RR FILES)
   1 - EXIT TO ASCL(1)

REPORT/DATA OUTPUT OPTION: ([H]DWCRFEO1) >
```

Figure 20. Report/Data dumper options.

```
REPORT/DATA OUTPUT OPTION: ([H]DWCRFE01) > D <

OUTPUT OPTIONS: ([H]TLBN) > T <

FILENAME ISCN.LD    15 : 47     7 / 23 / 1981

     SYSTEM: GSC0                          ANALYST: DPR
     ANALYTE: CO                           PARMETH: 224YZ02
     RUN FILE NAME: 0000000.RF             RF CREATED: 81/07/23 15:39
     INSTRUMENT: PE5000R562                UNITS: UG/L
     SEQUENCING:                           DATA ACCESS:
     COMPUTATION:                          OUTPUT:
     RESIDENT PGM: ISCN.04                 SOURCE PGM: ISCN.07
     HIGH STD:  120                        HIGH ABS:  .286
     LOW STD:  0                           LOW ABS:  1.9E-02
     STD FIELD: +++#.##                    ABS FIELD: +#.####

     SAMPLES:  17                          RUN FILE RECORDS:  39
     CAL STDS:  7                          UNKNOWNS:  4
     CHECK STDS:  1                        INST BLANKS:  1
     LAB SPIKES:  1                        REAGENT BLANKS:  1
     DUPLICATES:  1                        CNTRL STDS:  1

 WHEEL   /    SAMPLE     /   REC    QC    SAMP      CONCENTRATIONS
  POS    /  IDENTIFIER   /   NO.   PNTR   TYPE    MASSES AND VOLUMES

    1     3                    2      0     9   1.000E+00   0.000E+00
    2     3   +0.000E+00       3      0     1   1.000E+00   0.000E+00
    3     3   +5.000E+00       4      0     1   1.000E+00   5.000E+00
    4     3   +1.000E+01       5      0     1   1.000E+00   1.000E+01
    5     3   +2.000E+01       6      0     1   1.000E+00   2.000E+01
    6     3   +3.000E+01       7     -1     1   1.000E+00   3.000E+01
    7     3   +5.000E+01       8      0     1   1.000E+00   5.000E+01
    8     3   +1.000E+02       9      0     1   1.000E+00   1.000E+02
    9     2          LRB      10      0     7   1.000E+00   0.000E+00
   10     2          LCM      16      0     6   0.000E+00   2.000E+01
   11     2 EMSL0134          23      0     2   1.000E-01   1.000E-01
   12     2 EMSL0135          24      0     2   1.000E-01   1.000E-01
   13     2 EMSL0136 LDX      25     -1     2   1.000E-01   0.000E+00
   14     2 EMSL0137          35      0     2   1.000E+00   0.000E+00
   15     2 EMSL0136 LD2      36     13     5   1.000E+00   0.000E+00
   16     2 EMSL0136 LSF      37     13     3   0.000E+00   2.000E+00
   17     3   +3.000E+01      39      6     4   1.000E+00   3.000E+01
```

Figure 21. Report/Data dumper option 'D' with resulting output.

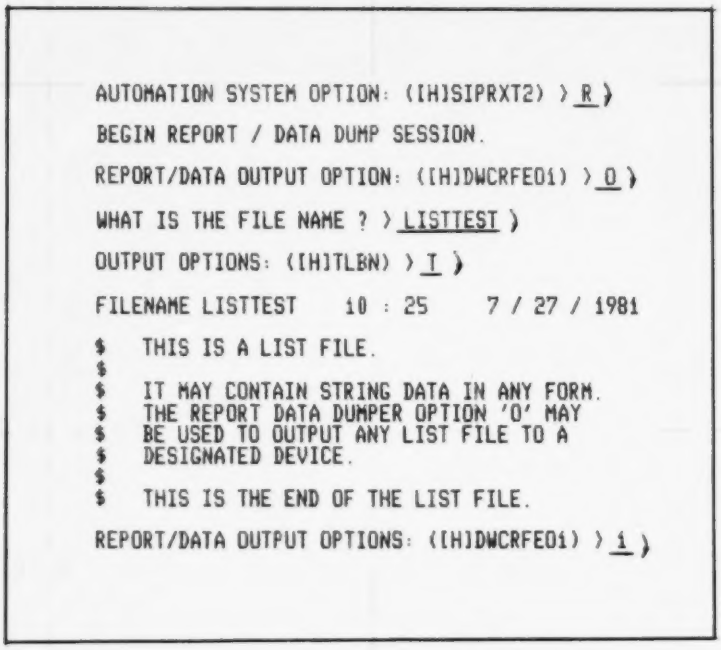


Figure 22. Report/Data dumper option 'O' with resulting output.

SECTION 4

STARTING AN AUTOMATED RUN - COMMAND 'S'

In order to make an automated RUN on a group of samples previously defined in a Run File, the 'S' command must be input at the ASCL. The analyst will be required to edit or accept numerous parameters prior to the actual measurement sequence. The input of this data as well as a detailed review of an automatic RUN sequence will be described in this section.

4.1 RUN FILE TRANSLATOR

4.1.1 Naming the Run File

The first step in performing an automated instrument RUN will be the naming of the Run File. If the Run File was developed through the SFC Master Command Level (SFCMCL) options[4] then the analyst will be required to type in the full name of the Run File which will be used during the RUN. If the Run File was created in the temporary Run File developer (Section 3.1), then a special default will be available for defining the Run File name. This name will always be '0000000.RF' and may be selected by pressing a RETURN instead of typing the Run File name. Figure 23 shows an example session for naming and translating a Run File.

4.1.2 Translation of the Run File

Once the Run File name has been specified and accepted, the computer will proceed to translate the Run File into a wheel pattern. Because of the nature of the standard calibration method, some wheel pattern sequences may not be allowed by the system and an error condition will arise. (See Section 4.1.3.) After the successful translation of a Run File, the computer will output the wheel pattern to the specified output device, as shown in Figure 23, and then proceed to the pre-RUN input phase described in Section 4.2.

4.1.3 Error Output Options for the Run File Translator

If the computer is not able to translate a Run File or if an improper sequence is detected then the system will terminate the RUN process and exit to the ASCL. The analyst will first be given an opportunity to output an error list to the appropriate output device. This list will have an error notation assigned to the record in the Run File which caused the termination. If the analyst is unable to determine why translation was unsuccessful he should consult with the system manager before trying to create another Run

```
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) > S ↵

WHAT IS THE RUN FILE NAME [0000000.RF] >

RUN FILE TRANSLATOR BEGINS (GSC0)

TRANSLATION COMPLETE.

RUN FILE CHECKS OK!

WHEEL PATTERN OUTPUT OPTIONS: ([H]TLBN)  > I ↵

WHEEL PATTERN FOR RUN FILE 0000000.RF    14 : 51    7 / 23 / 1981

WHEEL POS   1 /3                /  INSTR BLANK
WHEEL POS   2 /3   +0.000E+00 / INSTR CAL STD
WHEEL POS   3 /3   +5.000E+00 / INSTR CAL STD
WHEEL POS   4 /3   +1.000E+01 / INSTR CAL STD
WHEEL POS   5 /3   +2.000E+01 / INSTR CAL STD
WHEEL POS   6 /3   +3.000E+01 / INSTR CAL STD  (HAS QC)
WHEEL POS   7 /3   +5.000E+01 / INSTR CAL STD
WHEEL POS   8 /3   +1.000E+02 / INSTR CAL STD
WHEEL POS   9 /2           LRB / REAGENT BLANK
WHEEL POS  10 /2           LCM / LAB CNTRL STD
WHEEL POS  11 /2 EMSL0134     /UNKNOWN SAMPLE
WHEEL POS  12 /2 EMSL0135     /UNKNOWN SAMPLE
WHEEL POS  13 /2 EMSL0136 LDX /UNKNOWN SAMPLE  (HAS QC)
WHEEL POS  14 /2 EMSL0137     /UNKNOWN SAMPLE
WHEEL POS  15 /2 EMSL0136 LD2 / DUPLICATE  OF UNKNOWN SAMPLE IN POS  13
WHEEL POS  16 /2 EMSL0136 LSF /   SPIKE  OF   UNKNOWN SAMPLE IN POS  13
WHEEL POS  17 /3   +3.000E+01 /   CHECK  OF    INSTR CAL STD IN POS   6
```

Figure 23. Session to name and translate a Run File and output the wheel pattern to selected device.

```
WHAT IS THE RUN FILE NAME  > TEST.RF ↵

RUN FILE TRANSLATOR BEGINS (GSC0)
2 EMSL5679 LD3 P   XXXYZNN            UG/L                          ERROR

RUN FILE TEST.RF HAS AN ERROR!

ERROR LIST OUTPUT OPTIONS: ([H]TLBN) > I ↵

RUN FILE ERROR LIST FOR TEST.RF       11 : 54     7 / 15 / 1980

OK    1 80  7 15 11 29 GSC1  XXXYZNN DPR
OK    3   +0.000E+00 P   XXXYZNN             S
OK    3   +1.000E+01 P   XXXYZNN             S
OK    3   +2.000E+01 P   XXXYZNN             S
OK    3   +4.000E+01 P   XXXYZNN             S
OK    3   +7.500E+01 P   XXXYZNN             S
ERROR 2 EMSL5679 LD3 P   XXXYZNN            UG/L


UNSCHEDULED EXIT TO AUTOMATION COMMAND LEVEL.

AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) >
```

Figure 24. Translation error exit session with resulting output.

File. Figure 24 shows an example of the Error/Exit process.

4.1.4 Escape Sequencing

If an ESCape is pressed during the translation process, the session will be terminated and control will be transferred to the ASCL.

4.2 PRE-RUN INPUTS

Before the actual measurement portion of the RUN takes place, the analyst will be required to answer a number of questions. These questions will relate to overall RUN conditions as well as to specific sample variables. Each of the sessions will be described below in detail.

4.2.1 Preliminary Parameters

The analyst will be required to specify his initials, the analyte being measured, the units used in the analysis, and the instrument name. The only item which is restricted is the units field which must either be a 'UG/L' or an 'MG/L' for micrograms per liter or milligrams per liter respectively; or for solids, a 'UG/KG' or an 'MG/KG' for micrograms per kilogram or miligrams per kilogram respectively. The other three fields are non-critical and can remain blank. Figure 25 shows the input of these initial variables along with the entry of user specified comment fields. Note that the analyst need only press a RETURN to accept the last-used parameter or comment. Up to five comments may be input describing instrument conditions, sample source, or any other pertinent factors related to the RUN. Comment fields may be up to 72 characters long. The comment-field entry routine may be terminated by typing an 'X' at the comment prompt.

4.2.2 Spike Volume/Concentration and Mass Entry

If spiked samples are present in the wheel pattern, the analyst will be expected to define the volume of the spiking solution injected into each spiked sample, the original volume of the sample, the mass or concentration of the spike, and the dilution volume added to the sample, if any. These data are essential for the proper reporting of spiked sample concentration as well as quality control acceptability. An example spike-definition sesion is shown in Figure 26. If more than one spike is processed in this session, the default for volume and mass/concentration will be those used for the previous spiked sample.

The computer will be able to prompt for spike-mass or spike-concentration depending on whether the analyst inputs a zero-spike volume or a significant (non-zero) spike volume. If the spike volume is insignificant compared to the volume of sample, then only the mass of the spike and the volume of the unknown are required to fully define the spiked unknown.

Appendix B describes the spiking and dilution procedures and the computational algorithms involved in producing sample determinations and quality control assessments.

```
BEGIN PRE-RUN INPUT SESSION.

ANALYST'S INITIALS: [BJM] ) DPR )
ANALYTE MEASURED:   [TL] ) CO )
CONCENTRATION UNITS:[UG/L] ) )
INSTRUMENT NAME:    [CRA-90] ) PE5000R562 )

ENTER COMMENTS HERE:
COMMENT                ([COPY], X TO EXIT, OR TYPE NEW COMMENT)
NUMBER
  1   )[HEIGHT CALCULATIONS]
      )COMMENT FIELDS MAY BE USED TO ENTER )
  2   )PERTINANT INFORMATION ON ANYTHING )
  3   )FIVE 74 CHARACTER FIELDS MAY BE USED.)
  4   )X )
```

Figure 25. Pre-RUN input variables and comment entry session.

```
WHEEL POS  16 /2 EMSL0136 LSF / SPIKED UNKNOWN
VOLUME OF SPIKE: [0] ML ) )
MASS OF ADDED SPIKE: [0] UG ) 2 )
ORIGINAL SAMPLE VOLUME: [0] ML  ) 100 )
DILUTION VOLUME ADDED: [0] ML  ))
DILUTION FACTOR =  1

MISTAKES ON SPIKES ? ([N] OR Y)  ) )
```

Figure 26. Spike-sample parameter entry session.

```
DO YOU WANT TO MODIFY DILUTION FACTORS ? ([N] OR Y)  ) Y )

DILUTION OPTIONS: ([H]=WJX)  ) H )
   = - EQUAL DILUTIONS FOR ALL APPLICABLE SAMPLES
   W - DEFINE WHEEL POSITION OF SAMPLE TO BE DILUTED
   J - JUMP THROUGH LIST OF DILUTABLE SAMPLES
   X - EXIT SESSION / APPROVE DILUTIONS

DILUTION OPTIONS: ([H]=WJX)  ) W )

WHEEL POSITION OF SAMPLE TO BE DILUTED (0 TO EXIT)  ? 12 )
WHEEL POS  12 /2 EMSL0135     /CURRENT DILUTION FACTOR =  1
ORIGINAL SAMPLE VOLUME: [100] ML  ) )
DILUTION VOLUME ADDED: [0] ML  ) 100 )
DILUTION FACTOR =  2

WHEEL POSITION OF SAMPLE TO BE DILUTED (0 TO EXIT)  ? 0 )

DILUTION OPTIONS: ([H]=WJX)  ) X )
```

Figure 27. Preliminary dilution volume definition session.

4.2.3 Dilution Volume Entry

The analyst will be responsible for specifying which unknown samples have been diluted prior to the RUN and what the original volume and the added dilution volume is for each diluted sample. Three options will be available to the analyst for specifying sample dilution volumes. The first allows for a uniform dilution of all unknowns, the second allows the analyst to select the unknown to be diluted and the third asks the computer to cycle through each dilutable sample and allow the analyst to state whether the sample is to be diluted or not.

Figure 27 shows an example dilution session in which the analyst specifies the wheel position of the unknown and then defines the original sample volume and the dilution volume added. In all cases, the last used volumes will become the defaults for the next sample selected for dilution.

Movement out of the dilution session may only be accomplished with an input of 'X', indicating that dilutions are acceptable.

4.2.4 Editing or Accepting Check Standards Limits

The quality control limits used to evaluate instrument check standards measured during the RUN may be accepted as previously defined or may be edited as shown in Figure 28. Normally these parameters will remain consistent from one RUN to the next. (If no check standards are present in the wheel pattern, this session will be skipped by the computer).

The check standard is a very important quality control assessment of instrument stability but it has also become very important as a sequence verifier. For example, if samples are improperly placed in the wheel, a terrible check standard recovery will be the most obvious indication of the error.

4.2.5 Editing Initial RUN-Modes

The next parameters that must be defined prior to an automatic RUN are the initial RUN-modes. These modes include the preliminary computational mode, the preliminary sequencing mode, the absorbance data acquisition mode, and the preliminary output mode. It is necessary to define these modes prior to an automatic RUN so that no operator intervention will be required during the RUN. Figure 29 shows the full useage of the mode-edit function. Note that the default parameters are remembered from the last time the RUN-modes were defined and that a RETURN will accept the displayed default. It is very important that the analyst realize that the use of one mode does not exclude the use of another. For example, if the linear computation mode was selected prior to an automatic sequence and subsequent information suggested that the quadratic mode was more appropriate, then a simple mode switch operation (Section 5.6.1) would be all that was necessary to correct the miscalculation.

If the RUN is being performed in the semiautomatic sequencing mode the

```
EDIT CHECK STANDARD LIMITS ? ([N] OR Y) > Y ↵

   UPPER CONTROL LIMIT   [115]) ↵
   UPPER WARNING LIMIT   [110]) ↵
   MEAN                  [100]) ↵
   LOWER WARNING LIMIT   [90]) ↵
   LOWER CONTROL LIMIT   [85]) ↵
```

Figure 28. Check standard limit edit session.

```
LAST-USED INITIAL RUN-MODES: ([CR] FOR DEFAULT)
   COMPUTATIONAL: (HZLQCI) [QUADRATIC      ] > H ↵
     Z - FORCED ZERO LINEAR FIT
     L - REGULAR LINEAR FIR
     Q - QUADRATIC FIT
     C - CUBIC FIT
     I - INTERPOLATION
   COMPUTATIONAL: (HZLQCI) [QUADRATIC      ] > L ↵
   SEQUENCING:    (HFS)    [SEMI-AUTOMATIC ] > H ↵
     F - FULLY AUTOMATIC SEQUENCE
     S - SEMI-AUTOMATIC SEQUENCE (YOU CONTROL MEASUREMENTS)
   SEQUENCING:    (HFS)    [SEMI-AUTOMATIC ] > F ↵
     REPLICATES: [ 1 ] >
   DATA ACCESS:   (HMA)    [AUTOMATIC] > H ↵
     M - MANUAL OR KEYBOARD ENTRY OF ABSORBANCE DATA
     A - AUTOMATIC COMPUTER ACCESS OF ABSORBANCE DATA
   DATA ACCESS:   (HMA)    [AUTOMATIC] > M ↵
   OUTPUT REPORT: (HTLBN)  [BOTH TERM & LPT] > H ↵
     T - TERMINAL ONLY
     L - LINEPRINTER ONLY
     B - BOTH TERMINAL AND LINEPRINTER
     N - NONE GENERATED
   OUTPUT REPORT: (HTLBN)  [BOTH TERM & LPT] > T ↵

MISTAKES ON INITIAL RUN MODES ? ([N] OR Y)  > N ↵
```

Figure 29. RUN-mode edit session.

pre-RUN mode definitions will not be as significant since they can be changed easily from Instrument RUN Command Level (IRCL) as described in Section 5.

If a fully automatic sequence is to be performed, the analyst should be aware of the fact that the system can handle up to six absorbance measurements per sample.

4.2.6 Exiting to the Instrument Checkout Module

Once the pre-RUN input variables have been specified the computer will shift control to the instrument checkout module. (See Section 4.3 below.) The only exception to this will be the case where an analyst inputs absorbance data via the keyboard with the manual data acquisition mode. (See Section 5.6.2 below.) That is, no access will be made to the instrument checkout module if no instrument is being used to supply the absorbance data. In this case, control will be immediately transferred to the IRCL (Section 4.4) for either automatic or semiautomatic sequencing.

4.2.7 Escape Sequencing

If an ESCape is pressed at any time during the pre-RUN session, the following prompt will appear on the user terminal:

QUIT OR RESTART PRE-RUN INPUTS? ([Q] OR R) >

If a 'Q' is typed the computer will transfer control to the ASCL. If 'R' is typed, the computer will recycle to the beginning of the pre-RUN input session. (See Section 4.2.1 above.)

4.3 INSTRUMENT CHECKOUT

The instrument checkout module will be available to the analyst just prior to starting an automatic sequence. The checkout module has been included at this location in order to allow the analyst to verify instrument conditions just prior to an automated sequence and to allow for the validation of chosen timing parameters in those cases where an analoginterfaced instrument is being used. Hollow cathode lamp levels may be checked, test injections may be made, and crude peak plots may be generated to certify proper system operation.

Section 3.2 describes the usage of this module for both analog-interfaced and ASCII interfaced instruments.

If an escape is pressed during the checkout session, it is assumed that the analyst has encountered problems and wishes to abort the RUN. In this case control will be transferred to the ASCL.

4.4 AUTOMATIC RUN OF SAMPLE WHEEL

If the analyst selected the fully-automatic sequencing mode in the

pre-RUN mode selection session (Section 4.2.5) then the computer will start acquiring data automatically from the instrument as soon as the instrument checkout session has been completed. Unless the sequence is interrupted by the analyst or communications between instrument and computer are impaired, the system will collect and process data on all of the samples in the wheel and generate reports to the preselected output device. This section describes the type of output which may be expected during the course of an automatic RUN and it also describes analyst activities which may be performed during the sequence to insure the quality of sample determinations.

4.4.1 Measurement of Standards and the Display of Data

Normally calibration standards are located at the beginning of a sample wheel pattern. The computer will acquire and reduce absorbance data on each of the calibration standards. Figure 30 shows an example of standard measurement results as acquired and displayed to the user terminal.

4.4.2 Calibration Reporting and Off-Line Plotting

When the last of the standards is measured, the computer will perform a mathematical calibration of the standards and produce at least one equation relating concentration to measured absorbance. A calibration report will be generated to the preselected output device showing the results of the calibration. An example report is shown in Figure 31. Note that the equation on the report corresponds to the computational mode selected by the analyst in the pre-RUN input parameter session described in Section 4.2.5 above.

One very important parameter will be available on the calibration report. The fitting error will tell the analyst how well the selected computational mode fits the actual response of the standards. Normally an error or 10% or less is adequate and 5% or less is good. A special feature has been included in the system which will alert the analyst to a potentially erroneous calibration. If the fitting error exceeds 25% for a forced-zero linear fit, a cautionary message will be displayed to the analyst but the automatic sequence will continue unimpaired. The analyst will be required to review the calibration report (and plots) very carefully if the error condition arises during calibration.

A special parallel or off line plotting capability will be available in laboratories which have the appropriate graphics display device. By logging onto the graphics terminal with the proper plot-account name, the user can obtain a plot of any of the curves generated during the calibration of the standards. The calibration must be completed before this feature will be available to the analyst and the plot generation session will have no effect on the continuation of the automatic sequence.

The plot session itself will operate identically to the calibration plot module described in Section 3.3.1 above with the exception that, at the end of the plot session, the user will be automatically logged off of the plotting device to make way for other users.

```
MEAS POS   1 /3                  /  INSTR BLANK
  MEASURED ABS ) .000
  AVE.ABS=  0   NO.REPS =  1
  1 ) 0

MEAS POS   2 /3   +0.000E+00 / INSTR CAL STD
  MEASURED ABS ) .001
  AVE.ABS=  .001   NO.REPS =  1
  1 ) .001

MEAS POS   3 /3   +5.000E+00 / INSTR CAL STD
  MEASURED ABS ) .019
  AVE.ABS=  1.9E-02   NO.REPS =  1
  1 ) 1.9E-02

MEAS POS   4 /3   +1.000E+01 / INSTR CAL STD
  MEASURED ABS ) .024
  AVE.ABS=  2.4E-02   NO.REPS =  1
  1 ) 2.4E-02

MEAS POS   5 /3   +2.000E+01 / INSTR CAL STD
  MEASURED ABS ) .054
  AVE.ABS=  5.4E-02   NO.REPS =  1
  1 ) 5.4E-02

MEAS POS   6 /3   +3.000E+01 / INSTR CAL STD  (HAS QC)
  MEASURED ABS ) .090
  AVE.ABS=  9E-02   NO.REPS =  1
  1 ) 9E-02

MEAS POS   7 /3   +5.000E+01 / INSTR CAL STD
  MEASURED ABS ) .143
  AVE.ABS=  .143   NO.REPS =  1
  1 ) .143

MEAS POS   8 /3   +1.000E+02 / INSTR CAL STD
  MEASURED ABS ) .286
  AVE.ABS=  .286   NO.REPS =  1
  1 ) .286

MULTIPLE CURVE REGRESSION BEGINS

MULTIPLE CURVE REGRESSION COMPLETED
```

Figure 30. Calibration standard measurements and corresponding terminal output.

CALIBRATION STANDARD'S TABLE

MODE: LINEAR
NUMBER OF STDS: 7
FITTING ERROR: 4.30 %
TIME: 15:14 ON 7/23/1981
HIGHEST STD: 100
LOWEST STD: 5

EQUATION: CONC=-1.05E-01+3.50E+02*ABS

WHEEL POS		STANDARD IDENTIFIER	NO. REPS	AVE ABS	CONC EXPECT	CONC FOUND	DIFF
2	3	+0.000E+00	1	+0.0010	+0.00	+0.24	-0.24
3	3	+5.000E+00	1	+0.0190	+5.00	+6.54	-1.54
4	3	+1.000E+01	1	+0.0240	+10.00	+8.29	+1.71
5	3	+2.000E+01	1	+0.0540	+20.00	+18.78	+1.22
6	3	+3.000E+01	1	+0.0900	+30.00	+31.36	-1.36
7	3	+5.000E+01	1	+0.1430	+50.00	+49.90	+0.10
8	3	+1.000E+02	1	+0.2860	+100.00	+99.90	+0.10

Figure 31. Standard calibration report.

4.4.3 Sample Measurement, Reporting, and Quality Control Evaluation

Once a calibration equation has been developed, the computer will be able to compute sample concentrations for the remainder of the samples in the wheel. Figure 32 shows this output as it will be generated to the user terminal. Note that various forms of output will be produced depending on the type of sample measured. Any problems detected during the sequence will generate an audible response by the terminal. Normally, these "beeps" will not require any operator intervention unless a check standard measures out of control. In this case, the analyst will be responsible for aborting or continuing the run. (See Section 4.4.5 for a description of this function.) Only under exceptional conditions will the computer terminate an automatic sequence. This will happen only when an ASCII interfaced instrument begins outputting distorted or unrecognizable information or when an analog interfaced instrument produces an unrecognizable peak data array. If the RUN is aborted by the computer, a series of "beeps" will be generated to the terminal and control will be transferred to the IRCL.

4.4.4 Status Report Generation

After the last sample in the wheel has been measured for the last time in a fully automatic sequence, the computer will generate a status report to the pre-specified output device and will transfer control to the IRCL. This report will contain essentially the same information which was generated to the user terminal during the measurement process but will also contain pertinent RUN information and calibration curve data used in the determination process. Figure 33 shows an example interim status report generated by the computer after a fully automatic sampling sequence.

After the report is generated to the preselected output device, the computer will transfer control to the IRCL. (See Section 5.)

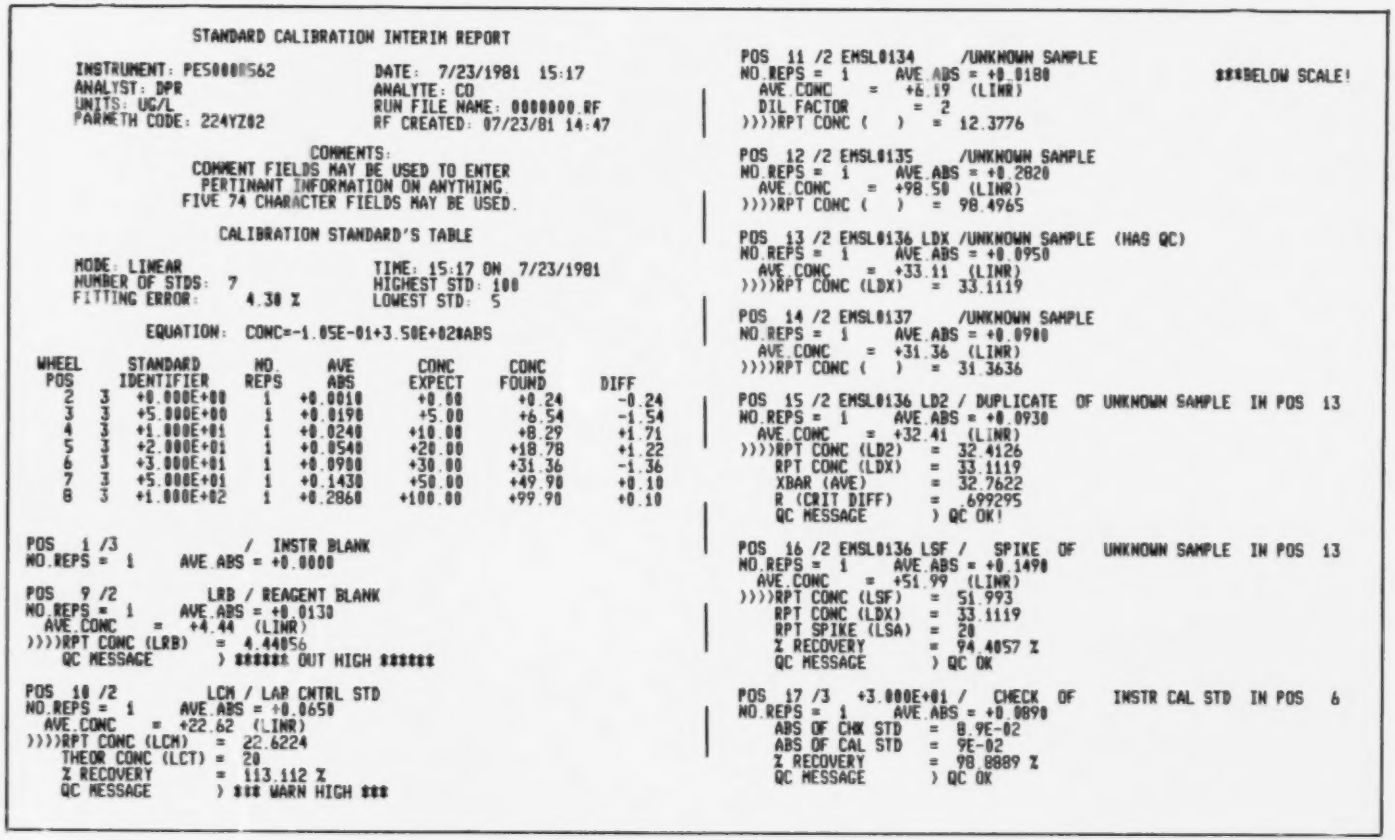
4.4.5 Escape Sequencing

If the analyst presses the ESCape key at any time during a fully automatic sampling sequence, the computer will transfer control to the IRCL. In some cases, the computer will finish the task it is working on before it acknowledges the ESCape and transfers control to the user.

Sometimes the termination of an automatic sequence through the ESCape mechanism will cause data to be stranded in the buffer box as in the case of ASCII interfaced instrument systems. The IRCL measure command ('M') described in Section 5.1 will be useful for recovering stranded data.

```
MEAS POS   9 /2          LRB / REAGENT BLANK
  MEASURED ABS )
  MEASURED ABS ) .065
  CALC CONC    =  22.6224   AVE.ABS=  6.5E-02   NO.REPS =  1
  1 ) 6.5E-02
  CURRENT CONC      =  22.6224 (LINR)
    RPT CONC (LCM)  =  22.6224
    THEOR CONC (LCT) =  20
    % RECOVERY      =  113.112 %
    QC MESSAGE      ) WARN HIGH!

MEAS POS  11 /2 EMSL0134     /UNKNOWN SAMPLE
  MEASURED ABS ) .018    OFF SCALE LOW!
  CALC CONC    =  6.18882   AVE.ABS=  1.8E-02   NO.REPS =  1
  1 ) 1.8E-02
  CURRENT CONC      =  6.18882 (LINR)
  DIL FACTOR        =  2
    RPT CONC (   )  =  12.3776

MEAS POS  12 /2 EMSL0135     /UNKNOWN SAMPLE
  MEASURED ABS ) .282
  CALC CONC    =  98.4965   AVE.ABS=  .282   NO.REPS =  1
  1 ) .282
  CURRENT CONC      =  98.4965 (LINR)
    RPT CONC (   )  =  98.4965

MEAS POS  13 /2 EMSL0136 LDX /UNKNOWN SAMPLE  (HAS QC)
  MEASURED ABS ) .095
  CALC CONC    =  33.1119   AVE.ABS=  9.5E-02   NO.REPS =  1
  1 ) 9.5E-02
  CURRENT CONC      =  33.1119 (LINR)
    RPT CONC (LDX)  =  33.1119

MEAS POS  14 /2 EMSL0137     /UNKNOWN SAMPLE
  MEASURED ABS ) .090
  CALC CONC    =  31.3636   AVE.ABS=  9E-02   NO.REPS =  1
  1 ) 9E-02
  CURRENT CONC      =  31.3636 (LINR)
    RPT CONC (   )  =  31.3636

MEAS POS  15 /2 EMSL0136 LD2 / DUPLICATE  OF UNKNOWN SAMPLE  IN POS  13
  MEASURED ABS ) .093
  CALC CONC    =  32.4126   AVE.ABS=  9.3E-02   NO.REPS =  1
  1 ) 9.3E-02
  CURRENT CONC      =  32.4126 (LINR)
    RPT CONC (LD2)  =  32.4126
    RPT CONC (LDX)  =  33.1119
    XBAR (AVE)      =  32.7622
    R (CRIT DIFF)   =  .699295
    QC MESSAGE      ) QC OK!

MEAS POS  16 /2 EMSL0136 LSF /   SPIKE  OF   UNKNOWN SAMPLE  IN POS  13
  MEASURED ABS ) .149
  CALC CONC    =  51.993   AVE.ABS=  .149   NO.REPS =  1
  1 ) .149
  CURRENT CONC      =  51.993 (LINR)
    RPT CONC (LSF)  =  51.993
    RPT CONC (LDX)  =  33.1119
    RPT SPIKE (LSA) =  20
    % RECOVERY      =  94.4057 %
    QC MESSAGE      ) QC OK

MEAS POS  17 /3   +3.000E+01 /   CHECK  OF    INSTR CAL STD  IN POS   6
  MEASURED ABS ) .089
  AVE.ABS=  8.9E-02   NO.REPS =  1
  1 ) 8.9E-02
    ABS OF CHK STD  =  8.9E-02
    ABS OF CAL STD  =  9E-02
    % RECOVERY      =  98.8889 %
    QC MESSAGE      ) QC OK
```

Figure 32. Unknown and quality control sample measurement and corresponding terminal output.



```
                    STANDARD CALIBRATION INTERIM REPORT

    INSTRUMENT: PES0000562              DATE:  7/23/1981  15:17
    ANALYST: DPR                        ANALYTE: CO
    UNITS: UG/L                         RUN FILE NAME: 0000000.RF
    PARMETH CODE: 224YZ02               RF CREATED: 07/23/81 14:47

                              COMMENTS:
                    COMMENT FIELDS MAY BE USED TO ENTER
                     PERTINANT INFORMATION ON ANYTHING.
                    FIVE 74 CHARACTER FIELDS MAY BE USED.

                        CALIBRATION STANDARD'S TABLE

    MODE: LINEAR                        TIME: 15:17 ON  7/23/1981
    NUMBER OF STDS:  7                  HIGHEST STD: 100
    FITTING ERROR:      4.30 %          LOWEST STD:  5

              EQUATION:  CONC=-1.0SE-01+3.50E+02*ABS

 WHEEL      STANDARD      NO.      AVE       CONC      CONC
  POS     IDENTIFIER     REPS      ABS     EXPECT    FOUND      DIFF
    2   3  +0.000E+00     1    +0.0010     +0.00      +0.24     -0.24
    3   3  +5.000E+00     1    +0.0190     +5.00      +6.54     -1.54
    4   3  +1.000E+01     1    +0.0240     +10.00     +8.29     +1.71
    5   3  +2.000E+01     1    +0.0540     +20.00     +18.78    +1.22
    6   3  +3.000E+01     1    +0.0980     +30.00     +31.36    -1.36
    7   3  +5.000E+01     1    +0.1430     +50.00     +49.90    +0.10
    8   3  +1.000E+02     1    +0.2860     +100.00    +99.90    +0.10

POS   1 /3              /  INSTR BLANK
NO.REPS =  1      AVE.ABS = +0.0000

POS   9 /2           LRB / REAGENT BLANK
NO.REPS =  1      AVE.ABS = +0.0130
  AVE.CONC    =   +4.44  (LINR)
))))RPT CONC (LRB)    =  4.44856
    QC MESSAGE         ) ****** OUT HIGH ******

POS  10 /2           LCM / LAB CNTRL STD
NO.REPS =  1      AVE.ABS = +0.0650
  AVE.CONC    =  +22.62  (LINR)
))))RPT CONC (LCM)    =  22.6224
    THEOR CONC (LCT) =  20
    % RECOVERY         =  113.112 %
    QC MESSAGE         ) *** WARN HIGH ***

POS  11 /2 EMSL0134      /UNKNOWN SAMPLE
NO.REPS =  1      AVE.ABS = +0.0180                    ***BELOW SCALE!
  AVE.CONC    =   +6.19  (LINR)
  DIL FACTOR          =  2
))))RPT CONC (    )    =  12.3776

POS  12 /2 EMSL0135      /UNKNOWN SAMPLE
NO.REPS =  1      AVE.ABS = +0.2820
  AVE.CONC    =  +98.50  (LINR)
))))RPT CONC (    )    =  98.4965

POS  13 /2 EMSL0136 LDX /UNKNOWN SAMPLE  (HAS QC)
NO.REPS =  1      AVE.ABS = +0.0950
  AVE.CONC    =  +33.11  (LINR)
))))RPT CONC (LDX)     =  33.1119

POS  14 /2 EMSL0137      /UNKNOWN SAMPLE
NO.REPS =  1      AVE.ABS = +0.0900
  AVE.CONC    =  +31.36  (LINR)
))))RPT CONC (    )    =  31.3636

POS  15 /2 EMSL0136 LD2 /  DUPLICATE  OF UNKNOWN SAMPLE  IN POS  13
NO.REPS =  1      AVE.ABS = +0.0930
  AVE.CONC    =  +32.41  (LINR)
))))RPT CONC (LD2)     =  32.4126
    RPT CONC (LDX)     =  33.1119
    XBAR (AVE)         =  32.7622
    R (CRIT DIFF)      =  .699295
    QC MESSAGE         ) QC OK!

POS  16 /2 EMSL0136 LSF /   SPIKE  OF   UNKNOWN SAMPLE  IN POS  13
NO.REPS =  1      AVE.ABS = +0.1490
  AVE.CONC    =  +51.99  (LINR)
))))RPT CONC (LSF)     =  51.993
    RPT CONC (LDX)     =  33.1119
    RPT SPIKE (LSA)    =  20
    % RECOVERY         =  94.4057 %
    QC MESSAGE         ) QC OK

POS  17 /3   +3.000E+01 /   CHECK  OF    INSTR CAL STD  IN POS   6
NO.REPS =  1      AVE.ABS = +0.0890
    ABS OF CHK STD     =  8.9E-02
    ABS OF CAL STD     =  9E-02
    % RECOVERY         =  98.8889 %
    QC MESSAGE         ) QC OK
```

Figure 33. Interim RUN status report.

SECTION 5

INSTRUMENT RUN COMMAND LEVEL OPTIONS

The Instrument RUN Command Level (IRCL) is the hub of this automation system. Its level number is 2 and it may be accessible with that number from the adjacent levels ASCL (1) and FCL (3) as described in Sections 5.8 and 5.9 below. The IRCL is capable of performing most system functions on command. Each function and corresponding command will be described in detail in this section. Figure 34 shows the format of the IRCL command line and the associated option list as generated by the help command.

5.1 MEASURE SAMPLE - COMMAND 'M'

In the semi-automatic mode, a sample may be measured using the 'M' command. An example of this command is shown in Figure 35. In order for this command to operate successfully, the analyst will be required to supply the computer with the wheel position of the next sample to be measured. The computer will know what type of sample is in the chosen position and will thus be capable of generating the proper results. It is the analyst's responsibility to make sure that the real sample being measured corresponds with the position specified in the command line.

The 'M' command will be useful for remeasuring samples that produce suspect results. The 'M' command may also be used to sequence through an entire sample wheel pattern. This would be a somewhat unproductive usage of the system but it may be necessary in those cases where no sample changer is available and injections are performed by hand.

5.2 SAMPLE EDITING - COMMAND 'E'

Sample data may be edited from the IRCL using the 'E' command. To "edit" sample data is defined as: the deletion of a single absorbance value maintained on a particular sample (S (POS)); the purge or erasure of all absorbance data maintained on a number of contiguous samples (P); or the modification of dilution volumes maintained on a particular sample (D(POS)) and the subsequent deletion of absorbance values maintained on the sample and associated QC samples.

Figure 36 shows the options available through the 'E' command. Each option will be described below but it should be noted that all edit command strings may be cascadable. This means that if the analyst inputs an 'ES13' at IRCL, the computer will perform intermediary steps without prompting the analyst for more information. Conversely, the analyst could input an 'E'

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) H ↓
   M[POS]  MEASURE SAMPLE IN POS
   C       CALIBRATE STANDARDS
   R       REPORT STATUS
   E#[POS] EDIT ABS OR DILUTION DATA (S[POS],P,D[POS])
   3       ACCESS FINISH COMMAND LEVEL(3)
   A       START AUTOMATIC RUN-SEQUENCE (WITH POS/REPS)
   U#      USER EDIT OF RUN-MODES (K,D,O)
   P#      PLOT CALIBRATION OR PEAK DATA (C OR P)
   1       EXIT IRCL(2) TO ASCL(1)

INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  )
```

Figure 34. Instrument RUN Command level (IRCL) options.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) M12 ↓

MEAS POS  12 /2 EMSL0135     /UNKNOWN SAMPLE
  MEASURED ABS ) .289   OFF SCALE HIGH!
  CALC CONC    =  100.944   AVE.ABS=  .2855   NO.REPS =  2
  1 ) .282    2 ) .289
  CURRENT CONC      =  99.7203 (LINR)
    RPT CONC (   )   =  99.7203
```

Figure 35. Measure sample - IRCL command 'M'.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) E ↓

SAMPLE EDIT OPTION: ([H]SPD2) ) H ↓
   S[POS]- SELECTIVE DELETION OF REPLICATE ABSORBANCE
   P     - PURGE DATA FOR SPECIFIED SAMPLES
   D[POS]- CHANGE DILUTION VOLUMES
   2     - RETURN TO IRCL(2)
```

Figure 36. Edit sample data - IRCL command 'E'.

and be prompted as in Figure 37. He may then input an 'S' and be further prompted to specify the wheel position. (See Section 5.2.1 below.)

5.2.1 Selective Deletion of Sample Absorbance Data - 'ES(POS)'

The edit option 'S' will allow the analyst to delete one or more replicate absorbance values for a specified sample in the wheel pattern. Figure 36 shows the usage of this option. Note that the wheel position of the sample in question must be typed immediately after the 'S' or the computer will prompt the analyst for the wheel position. Once the positon is specified, the computer will display all relevant data on the sample before asking which replicate is to be deleted. It will not be necessary for the analyst to delete any data on the sample, thus making the command useful as a sample previewer. The computer will keep asking which replicate is to be deleted until the analyst presses RETURN or types a '0'. If there are no replicates left to be deleted, the computer will automatically exit the session.

After a selective-edit session is complete, the computer will shift control back to the edit option line.

5.2.2 Purging Absorbance Data for Multiple Samples - 'EP'

The edit option 'P' will allow the analyst to erase absorbance data on one or more sequential samples. Figure 38 shows the usage of this option. Once the purge option is invoked the computer will prompt the analyst to input the first and last positions of the samples being purged. After the positions have been validated, the computer will erase all absorbance data maintained on the affected samples.

After the purge session is completed, control will shift back to the edit option line.

5.2.3 Modify Dilution Volumes for a Selected Sample - 'ED(POS)'

The edit option 'D' will allow an analyst to modify the original and dilution volumes maintained on a sample prior to the actual dilution and remeasurement of the sample. If a sample measured off scale during an automatic RUN, the analyst may choose to dilute and remeasure the sample "on-the-spot" in order to obtain unqualified sample results. Figure 39 shows the usage of the edit 'D' option. Note that the position of the sample to be diluted must be entered immediately after the 'D' or the computer will prompt the analyst for the wheel position. (Note that only unknown samples may be diluted.) The analyst must then specify the original sample volume and the volume of added dilution solution. After receiving the volume information, the computer will perform one or more of the following steps: update the volume information for the specified sample; delete current absorbance data maintained on the sample; and delete current absorbance data on any quality control spike or duplicate associated with the sample being diluted.

```
SAMPLE EDIT OPTION: ([H]SPD2) ) 2 )

INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) E )

SAMPLE EDIT OPTION: ([H]SPD2) ) S )

SAMPLE POSITION ) 10 )

EDIT POS  10 /2          LCM / LAB CNTRL STD
  AVE.ABS=  6.5E-02   NO.REPS =  1
  1 ) 6.5E-02
  CURRENT CONC      =  22.6224 (LINR)
    RPT CONC (LCM)  =  22.6224
    THEOR CONC (LCT) =  20
    % RECOVERY      =  113.112 %
    QC MESSAGE      ) WARN HIGH!

DELETE REPLICATE ABS NUMBER ([0] TO EXIT) ) 1 )
  AVE.ABS=  0   NO.REPS =  0

SAMPLE EDIT OPTION: ([H]SPD2) ) 2 )
```

Figure 37. Edit option 'S' - selective deletion of sample data.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) E )

SAMPLE EDIT OPTION: ([H]SPD2) ) P )

FIRST POSITION   [1]  ) )
LAST POSITION    [17]  ) 9 )

SAMPLE EDIT OPTION: ([H]SPD2) ) 2 )

YOU HAVE MODIFIED STANDARD ABS DATA.
PLEASE RECALIBRATE AS SOON AS POSSIBLE
```

Figure 38. Edit option 'P' - purge sample data.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) E )

SAMPLE EDIT OPTION: ([H]SPD2) ) D )

SAMPLE POSITION ) 12 )

DILT POS  12 /2 EMSL0135     /UNKNOWN SAMPLE
  AVE.ABS=  .2855   NO.REPS =  2
  1 ) .282    2 ) .289
  CURRENT CONC      =  99.7203 (LINR)
    RPT CONC (   )  =  99.7203

MODIFY DILUTION VOLUMES ? ([N] OR Y) ) Y )
   ORIGINAL SAMPLE VOLUME: [0] ML ) 100 )
   DILUTION VOLUME ADDED: [0] ML ) 100 )
DILUTION FACTOR =  2

PLEASE FOLLOW DILUTION PROCEDURES
AND REMEASURE SAMPLES NOTED ABOVE.

SAMPLE EDIT OPTION: ([H]SPD2) ) 2 )
```

Figure 39. Edit option 'D' - modify dilution volumes.

After the dilution volume edit session is complete, control will shift back to the edit option line.

5.2.4 Exiting the Option Module

The edit module can only be exited through the command '2' which will transfer control to level 2 or the IRCL. This will allow multiple usage of the purge, delete, and dilute options for any single invocation of the 'E' command from the IRCL.

5.2.5 Escape Sequencing

If the analyst presses the ESCape key at any time in the edit module, control will shift to the edit option line. If an ESCape key is pressed during a sample data purge, the analyst will have to search through the affected samples to determine how far the purge progressed before being interrupted.

5.3 CALIBRATING STANDARDS - COMMAND 'C'

Standards may be calibrated at any time from IRCL with the 'C' command. It will not be necessary to recalibrate the standards unless standard absorbance values have been deleted by editing, or additional measurements are taken on previously calibrated standards. Section 5.6.1 should be consulted if the analyst simply wishes to change the computational mode used to compute sample concentrations.

When the 'C' command is input by the analyst, the computer will perform the calibration on all standard replicate measurements previously taken in the RUN. When calibration is complete, the computer will output a calibration report to the previously chosen output device (Figure 31). The output device may be changed as described in Section 5.6.3.

The analyst should take special note of the fact that the calibration report generated with the 'C' command only represents one of up to five equations that the computer generates during calibration. The analyst may change the computational mode at any time after calibration with the procedure described in Section 5.6.1.

During the calibration process, a plot file will be generated containing all of the information necessary to produce calibration plots on the appropriate graphics display device. Calibration plot applications are described in Sections 3.3.1, 4.4.2, and 5.7.

The calibration curve plots will give the analyst the best information regarding the type of computational mode needed to make accurate determinations. However, on those systems where no plotting facility is available, the analyst will have to depend on the calibration report to supply this information. The fitting error will be a primary factor in the acceptance or rejection of a particular computational mode. In addition the residuals may show possible outliers or indicate unexpected curvature over the concentration range.

The system has built in precautions in order to compensate for unacceptable mode selections made by the analyst. For example, the computer will not allow an analyst to calibrate two standards with a second degree curve. It will always be recommended that an analyst measure a sufficient number of standards in order to avoid mathematical errors inherent in the calibration process. Appendix A indicates the number of standards recommended for each of the four major computational modes.

Interpolation is treated somewhat differently from the other computational modes. (Appendix A describes the interpolation process.) A problem arises when the absorbance falls below that measured for the lowest calibration standard or above that measured for the highest calibration standard. In these cases the system will revert to the forced-zero linear equation to compute sample concentrations.

Once a calibration report has been generated, the computer will transfer control to the IRCL. If the ESCape key is pressed any time during calibration, the computer will ignore the interrupt and will complete the calibration and return to he IRCL. (Escaping calibration during an automatic sequence is described in Section 4.4.5 above).

5.4 REPORT RUN STATUS - COMMAND 'R'

The analyst may generate an interim status report on the RUN at any time from the IRCL by using the 'R' command. Figure 33 shows an example RUN status report. The report will be generated to the preselected output device. The output mode may easily be modified prior to report generation as described in Section 5.6.3 below.

If calibration has not been performed on the standards, then no calibration subreport will be furnished in the RUN status report. The analyst should note that the 'R' command can be used at any time at the IRCL and if no data is maintained on a particular sample then only the sample position and description will be output.

After the computer has generated the status report, control will be transferred to the IRCL. If the ESCape key is pressed any time during the report generation process, the computer will ignore the interrupt and will complete the task and return to the IRCL. (Escaping report generation during an automatic sequence is described in Section 4.4.5 above).

5.5 RESTART AN AUTOMATIC RUN SEQUENCE - COMMAND 'A'

An automatic sequence can be initiated at any time from IRCL with the use of the 'A' command. An example of the use of this command is shown in Figure 40. The analyst will be required to specify the starting wheel position and the number of replicate measurements to be taken on each sample.

The system will not allow the analyst to specify more than six replicates per sample, however, it will not be able to protect the analyst from making more than six total measurements on any given sample. If six

replicates are maintained on a sample and the analyst executes another measurement either with the 'M' command (Section 5.1) or through an automatic sequence, the computer will simply ignore the absorbance value acquired in the measurement. The analyst will have to delete one of the six replicates with the 'ES' command (Section 5.2.1) before another replicate absorbance will be accepted.

The operation of an automatic RUN sequence has been described in Section 4.4. The analyst should note that the default computation and output modes will be used in generating reports during the automatic sequence. These modes may be changed prior to the use of the 'A' command as described in Section 5.6 below.

5.6 RUN MODE MODIFICATION - COMMAND 'U'

The RUN-modes can be modified by the analyst from the IRCL with the 'U' command. Changes may be made in the computational mode ('UK#'), the data acquisition mode ('UD'), or the output mode ('UO#'). The commands may be cascaded or may be input one character at a time through multiple computer prompts. Figure 41 shows the options available to the analyst through the 'U' command. This section will describe each option and the corresponding suboption to the 'U' command.

5.6.1 Computational Mode Modification - 'UK#'

The analyst may modify the computational mode from the RUN-mode modification option line (Figure 41). If the analyst enters a 'K' the computer will prompt the analyst for the desired computational mode. The available suboptions are shown in Figure 42.

The computer will reject a particular analyst selected computational mode only if the number of standards in the wheel pattern was insufficient to produce the desired equation. In such cases, the analyst will be asked to select a lower order equation for computing sample concentrations.

After the computational mode has been modified, the computer will transfer control to the IRCL. Mode changes can be made repeatedly without affecting data maintined on the standards. The only affect of a mode change will be in the reported concentration values generated by the computer.

If the analyst presses the ESCape key at any time in the mode selection process, the computer will transfer control to the IRCL and await another command.

5.6.2 Data Acquisition Mode Modification - 'UD'

This system has been designed primarily to interface operating instruments to the lab automation computer. An additional feature has been included, however, which will allow the analyst to switch from a system which automatically collects and reduces absorbance data (from an on-line instrument), to a system which prompts the analyst for absorbance data directly from the keyboard. An example of the use of this option is shown

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  > A
NUMBER OF REPLICATES > 1
STARTING POSITION > 1

MEAS POS   1 /3                /  INSTR BLANK
  MEASURED ABS >
```

Figure 40. Restart automatic sequence - IRCL command 'A'.

```
MODE EDIT OPTIONS: ([H]KDO) > H
   K - CHANGE COMPUTATIONAL MODE (ZLQCI)
   D - CHANGE DATA ACQUISITION MODE
   O - CHANGE OUTPUT MODE (TLBN)
```

Figure 41. RUN-mode modification - IRCL command 'U'.

```
MODE EDIT OPTIONS: ([H]KDO) > K

COMPUTATIONAL MODE: ([H]ZLQCI) > H
   Z - FORCED ZERO LINEAR
   L - REGULAR LINEAR
   Q - QUADRATIC
   C - CUBIC
   I - INTERPOLATION

COMPUTATIONAL MODE: ([H]ZLQCI) > Q
```

Figure 42. RUN-mode option 'K' - modify computational mode.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  > U
MODE EDIT OPTIONS: ([H]KDO) > D
CHANGE FROM KEYBOARD TO COMPUTER ACQUISITION.
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  > U
MODE EDIT OPTIONS: ([H]KDO) > D
CHANGE FROM COMPUTER TO KEYBOARD ACQUISITION.
```

Figure 43. RUN-mode option 'D' - modify data acquisition mode.

in Figure 43. The transition can be made from computer controlled data acquisition to manual entry of sample absorbance data and back again with the 'D' option. If the analyst begins the RUN in the manual-entry mode, however, no transition will be allowed to the computer controlled data acquisition mode.

After the data acquisition mode has been modified, the computer will transfer control to the IRCL. Note that in the manual-entry mode, the analyst will be responsible for supplying sample absorbance data either through the 'M' command or during an automatic sequence. All computer responses to a manually entered absorbance value will be identical to those produced for computer collected data.

5.6.3 Output Mode Modification - 'UO#'

The analyst may change the output mode with the 'O' option as shown in Figure 44. The computer will output all major reports to the device or devices specified by the output mode. This mode will have been defined in the pre-RUN mode selection process as described in Section 4.2.5 above. The analyst may modify the mode at any time from the IRCL. Note that the modification of the output mode will in no way affect the sample-specific data generated to the user terminal during measure/edit operations.

After the output mode has been modified, the computer will transfer control to the IRCL. Subsequent output reports will be generated to the appropriate device.

If the analyst presses the ESCape key prior to the actual mode modification, control will be immediately transferred to the IRCL.

5.7 PLOT OPTIONS FROM THE IRCL - COMMAND 'P'

The plot options described in Section 3.3 are available to the analyst at the IRCL only in those cases where the user-terminal is an appropriate graphics display device. Both calibration and raw peak data plotting will be available under these circumstances. The analyst must follow the instructions described in Section 3.3. When the analyst has completed a plot session, the computer will transfer control to the IRCL and await a user command.

5.8 FINAL REPORT GENERATION - COMMAND '3'

At some point in the RUN, the analyst may decide to generate a final report. He may do so by using the '3' command at the IRCL. This will cause the computer to transfer control to system level 3, or the Finish Command Level (FCL). Section 6 of this manual will describe this command level in detail.

The analyst should note that the transition to the FCL will in no way impede the analyst's ability to reenter the IRCL and continue the process of measuring and editing sample data.

5.9 EXITING FROM IRCL TO ASCL - COMMAND '1'

The RUN may be terminated by entering a '1' at the IRCL. The results of this command will be the transfer of control to the ASCL at which point the analyst can exit the entire automation system. The system has been designed so that the analyst can return to the IRCL from ASCL with exactly the same status as when the IRCL was exited. This feature is detailed in Section 3.5.

Figure 45 shows an example of the use of the '1' command at the IRCL. The analyst should note that the only way to get to the ASCL from the IRCL is with the '1' command.

5.10 ESCAPE SEQUENCING

If the analyst presses the ESCape key at any time during the accomplishment of an IRCL function, the computer will return control to the IRCL. Sometimes it will finish its current task before returning to the IRCL as in the case of calibration or report generation. At most other times, return to IRCL will be immediate. The individual descriptions of IRCL commands should be consulted for more details regarding escape sequencing.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUPi)  ) U )

MODE EDIT OPTIONS: ([H]KDO) ) O )

OUTPUT OPTIONS: ([H]TLBN) ) H )
   T - TERMINAL
   L - LINEPRINTER
   B - BOTH TERMINAL AND LINEPRINTER
   N - NONE

OUTPUT OPTIONS: ([H]TLBN) ) B )
```

Figure 44. RUN-mode option 'O' - modify output mode.

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUPi)  ) 1 )
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) )
```

Figure 45. Exit to ASCL - IRCL command '1'.

SECTION 6

FINISH COMMAND LEVEL

Level 3 of the ISC system is called the Finish Command Level (FCL). Figure 46 shows the available FCL options. The purpose of the FCL is to produce a final report indicating the results of the RUN. This report may be in the form of an SFC Run Results File or a formatted (non-SFC) Final Report which may be delivered to a requestor. The generation of one of these types of reports will not interfere with the analyst's ability to generate the other. However, the Run Results File generator is specifically designed for those systems with an SFC interface and the Final Report generator is designed for non-SFC or research applications.

Two special prereport functions will be available to the analyst to facilitate reagent blank subtractions and RUN limit modification. The report and prereport functions will be described in detail in this section.

6.1 EDIT BDL/GTL LIMITS - 'L'

The system has been designed to flag any unknown sample result as below the determination level (BDL) if the sample concentration falls below the lowest non-zero calibration standard measured during the RUN. It will also flag any unknown sample result as greater than the analysis limit (GTL) if the sample concentration exceeds the concentration of the highest calibration standard measured during the RUN. The BDL or GTL result qualifiers will appear next to any unknown sample which violates the corresponding limit. (Reference 1 describes these qualifiers.)

The analyst will be given the opportunity to adjust these limits from the FCL with the use of the 'L' command. An example of the usage of this command is shown in Figure 47.

Normally, the 'L' command will be used to slightly expand the concentration range of the analysis. In fact, however, these limits may be expanded indefinitely. The analyst will be completely responsible for the proper maintenance of these levels as they relate to the RUN specifically, and as they relate to the analytical method generally.

6.2 DEFINE REAGENT BLANK LEVELS - 'B'

An analyst may measure reagent blanks during the course of a RUN in order to assess contamination levels introduced by method processing. The use of reagent blanks and control standards in determining reagent contamination is described in detail in Appendix A. If an analyst detects a

```
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  > 3 ↓

FINISH COMMAND LEVEL OPTIONS ([H]LBGF2) > H ↓
   L - MODIFY BDL/GTL LIMITS
   B - REAGENT BLANK ABSORBANCE SUBTRACTION
   G - GENERATE RUN RESULTS FILE (SFC)
   F - OUTPUT FINAL REPORT (NON-SFC)
   2 - EXIT TO ISCL(2)
```

Figure 46. Finish Command Level (FCL) options.

```
FINISH COMMAND LEVEL OPTIONS ([H]LBGF2) > L ↓

LOWER CONCENTRATION LIMIT [5UG/L] > 0 ↓
UPPER CONCENTRATION LIMIT [100UG/L] > 120 ↓
```

Figure 47. FCL option 'L' - edit BDL/GTL limits.

```
FINISH COMMAND LEVEL OPTIONS ([H]LBGF2) > B ↓

REAGENT BLANK ABSORBANCE ([CR] TO EXIT)  > .001 ↓
STARTING WHEEL POSITION [1] >
STOPPING WHEEL POSITION [17] >

REAGENT BLANK ABSORBANCE ([CR] TO EXIT)  >
```

Figure 48. FCL option 'B' - reagent blank subtraction.

uniform rise in absorbance signal levels due to sample preparation, the 'B' command may be used to compensate for the increased absorbance. An example of the usage of this command is shown in Figure 48.

Normally, all of the non-standard samples will be prepared identically for a given RUN. In this case, one reagent blank and one control standard will be sufficient to determine whether a uniform contamination has been introduced by the method. The analyst will be expected to review the RUN status report and use the FCL command 'B' to enter a reagent subtraction value. The analyst must specify the samples that are affected by the reagent subtraction. The specified absorbance value will be stored along with other sample data and will in no way distort actual sample absorbance values. The reagent subtractor will not affect output for standards, check standards, reagent blanks, or control standards.

Once the reagent subtractor has been specified, it will remain as part of the sample data base. The analyst can alter the value at any time or may erase it completely by entering a zero reagent subtractor for the entire wheel pattern.

In special cases, more than one reagent subtractor may be applied to a wheel pattern. If the first group of non-standard samples have been processed by method-A and the next group of non-standard samples have been processed by method-B, and corresponding reagent blanks and control standards have been similarly processed, then the analyst may enter two reagent subtractors for the corresponding samples of each group.

Extreme caution should be exercised in using the reagent subtractor mechanism. Subsequent report generation will show the affects of the adjusted sample concentrations and these values may be significantly different from those generated in the interim status report.

6.3 GENERATE SFC RUN RESULTS FILE - 'G'

In all SFC applications, the ultimate product of an automated RUN is the generation of a Run Results File. The analyst who is satisfied with the sample results shown in the interim status report may generate the final results to the Run Results File through FCL command 'G' as shown in Figure 49. The use of this command will simply supply results to the SFC Run File. Some results will be supplied in the form of absorbance values as in the case of standards and check standards, while the fields assigned to unknowns, spikes, duplicates, reagent blanks, and control standards will be filled with actual concentration values. No results will be supplied to a sample field if no replicate absorbance measurements are available for the sample.

The 'G' command will require no additional response from the analyst since the computer will automatically formulate the Run Results File and output it to the preselected output device. (See Section 5.6.3 for output mode modification.) The analyst may execute the 'G' command more than once. Old information will be erased and new information will be supplied

```
FINISH COMMAND LEVEL OPTIONS ([H]LBGF2) ) G )

RUN RESULTS FILE 0000000.RR    15 : 39     7 / 23 / 1981

1 81 07 23 15 39 GSC0  224YZ02 DPR
3                P    224YZ02 +0.000E+00 B
3   +0.000E+00 P    224YZ02 +1.000E-02 S
3   +5.000E+00 P    224YZ02 +1.900E-02 S
3   +1.000E+01 P    224YZ02 +2.400E-02 S
3   +2.000E+01 P    224YZ02 +5.400E-02 S
3   +3.000E+01 P    224YZ02 +9.000E-02 S
3   +5.000E+01 P    224YZ02 +1.430E-01 S
3   +1.000E+02 P    224YZ02 +2.860E-01 S
2           LRB P    224YZ02 +6.148E-02 UG/L
4 LRBL      UCL P    224YZ02 +3.000E+00 MM/DD/YY
4 LRBL      UWL P    224YZ02 +2.000E+00 MM/DD/YY
4 LRBL      AVE P    224YZ02 +0.000E+00 MM/DD/YY
4 LRBL      LWL P    224YZ02 -2.000E+00 MM/DD/YY
4 LRBL      LCL P    224YZ02 -3.000E+00 MM/DD/YY
2           LCM P    224YZ02 +2.340E+01 UG/L
2           LCT P    224YZ02 +2.000E+01 UG/L
4 LCST      UCL P    224YZ02 +1.150E+02 MM/DD/YY
4 LCST      UWL P    224YZ02 +1.100E+02 MM/DD/YY
4 LCST      AVE P    224YZ02 +1.000E+02 MM/DD/YY
4 LCST      LWL P    224YZ02 +9.000E+01 MM/DD/YY
4 LCST      LCL P    224YZ02 +8.500E+01 MM/DD/YY
2 EMSL0134      P    224YZ02 +7.448E+01 UG/L          SFCRECNO BKNUM
2 EMSL0135      P    224YZ02 +1.991E+02 UG/L          SFCRECNO BKNUM
2 EMSL0136 LDX P    224YZ02 +3.258E+01 UG/L          SFCRECNO BKNUM
4 LSPK      UCL P    224YZ02 +1.225E+02 MM/DD/YY
4 LSPK      UWL P    224YZ02 +1.150E+02 MM/DD/YY
4 LSPK      AVE P    224YZ02 +1.000E+02 MM/DD/YY
4 LSPK      LWL P    224YZ02 +8.500E+01 MM/DD/YY
4 LSPK      LCL P    224YZ02 +7.750E+01 MM/DD/YY
4 LDUP      A   P    224YZ02 +0.000E+00 MM/DD/YY
4 LDUP      B   P    224YZ02 +0.000E+00 MM/DD/YY
4 LDUP      C   P    224YZ02 +2.000E-02 MM/DD/YY
4 LDUP      D   P    224YZ02 +2.000E+00 MM/DD/YY
2 EMSL0137      P    224YZ02 +7.183E+01 UG/L          SFCRECNO BKNUM
2 EMSL0136 LD2 P    224YZ02 +3.151E+01 UG/L
2 EMSL0136 LSF P    224YZ02 +5.200E+01 UG/L
2 EMSL0136 LSA P    224YZ02 +2.000E+01 UG/L
3   +3.000E+01 P    224YZ02 +8.900E-02 CS
```

Figure 49. FCL option 'G' - Run Results File generation.

each time a Run Results File is generated. This will allow an analyst to reenter the IRCL, perform additional measure/edit functions, and regenerate an updated Run Results File from the FCL with the 'G' command.

6.4 GENERATE NON-SFC FINAL REPORT - 'F'

A non-SFC Final Report may be generated from the FCL with the 'F' command as shown in Figure 50. This report will contain the final concentrations for non-standard samples, the known theoretical values for spikes and control standards, and the method-limit and quality control limit violations for those samples requiring qualification.

The 'F' command will require no additional response from the analyst since the computer will automatically formulate the Final Report and output it to the preselected output device. The analyst may execute the 'F' command repeatedly. This will allow an analyst to reenter the IRCL, perform additional measure/edit functions, and reproduce an updated Final Report from the FCL with the 'F' command.

6.5 EXIT FROM FCL TO THE IRCL - '2'

The analyst may exit the FCL and transfer control to the IRCL by using the '2' command. This will move control from level 3 to level 2 of the system. This will be the only way to exit the FCL. After the analyst has completed all aspects of the RUN and generated the appropriate output, he must exit the FCL to the IRCL and then exit the IRCL to the ASCL before an exit can be made from the instrument automation system. Figure 51 shows the exit sequence from the FCL to the ASCL.

6.6 ESCAPE SEQUENCING

An ESCape may be pressed at any time during an FCL function. The computer will immediately transfer control to the FCL option line and await a user-command. Escaping a report generation process will not damage data or preclude the future use of any report generation command. However, the analyst should be aware of the fact that no valid report will be available if an ESCape is pressed during the report generation process.

```
FINISH COMMAND LEVEL OPTIONS ([H]LBGF2) ) F )


          FLAMELESS AA STANDARD CALIBRATION FINAL REPORT

    INSTRUMENT: PE5000R562              DATE:  7/23/1981  15:39
    ANALYST: DPR                        ANALYTE: CO
    UNITS: UG/L                         RUN FILE NAME: 0000000.RF
    PARMETH CODE: 224YZ02               RF CREATED: 07/23/81 15:39

 WHEEL      SAMPLE          SAMPLE      KNOWN SPIKE    VIOLATION
  POS     IDENTIFIER     CONCENTRATION  OR CONTROL     LIMIT     QC

   9              LRB    +6.148E-02                              OK
  10              LCM    +23.3971        +20.000                 OUTC
  11     EMSL0134        +74.4846
  12     EMSL0135        +199.085
  13     EMSL0136 LDX    +32.5835
  14     EMSL0137        +71.8315
  15     EMSL0136 LD2    +31.5061                                OK
  16     EMSL0136 LSF    +52.0043        +20.000                 OK
```

Figure 50. FCL option 'F' - Final Report generation.

```
FINISH COMMAND LEVEL OPTIONS ([H]LBGF2) ) 2 )
INSTRUMENT RUN OPTIONS: ([H]MCRE3AUP1)  ) 1 )
AUTOMATION SYSTEM OPTION: ([H]SIPRXT2) ) X )
NOW EXITING AUTOMATION SYSTEM.
```

Figure 51. Exit the automation system from the FCL.

SECTION 7

NOTES TO THE SYSTEM MANAGER

The ISC system is composed of up to 15 programs and as many as 13 data or list files (See Table 2). The majority of these programs and files are named with a prefix of 'ISCN' where:

I - Instrument identifier;
S - Standard;
C - Calibration;
N - Instrument number.

The actual directory names for the ISC system users will take the form ISCN so that a Flameless AA account identifer may be GSC1 while a Flame AA account name may be FSC2. The overall SFC system will dictate the directory name utilized on a particular ISC application.

All ISCN automation system programs will take the form ISCN.## where the '##' is an integer number from 00 to 12. Each program has a particular function or set of functions. Transportation between programs is performed by CHAINing and is invisible to the user.

The system utilizes a small file called INST.NM to determine the directory that it occupies. The AUTORUN program reads this file and displays the directory name. It then CHAINS to the Automation System Command Level router program (ISCN.00). INST.NM is a permanent sequential file and must be read with the LREAD command in order to obtain the appropriate directory name. If INST.NM is not available, the instrument system will note the error and exit the automation system.

Three important files will contain the main body of information relevant to a given RUN. These include the sample identifer file (ISCN.ID), the sample data file (ISCN.DF), and the system transfer file (ISCN.TR). In addition, ISCN.L# and ISCN.P# files will be formed during normal system operations and these files represent list and plot files, respectively. These files are normally output or accessed at appropriate times during a RUN, however, system utilities are available to the system manager or analyst for accessing these files outside the context of a RUN. (See Section 3.)

Only two automation system programs do not follow the ISCN naming conventions. These are AUTORUN which falls within the framework of the overall SFC system[4] and INITIALIZE.2 which has been especially formulated for

calibration curve plotting. A special plot-account name will be available to the ISC system user in order to utilize the laboratory plot-terminal for the generation of calibration curves. Examples of this special account name are FSP1 and GSP2. An analyst logging on to a plot terminal with the plot account name will immediately invoke the INITIALIZE.2 program. This program will CHAIN ISCN.08 which is the calibration curve plotting program. Once the plot data has been reviewed, the plot program will automatically log off so that other analysts may avail themselves of the graphics device. Special command lines in BASIC.ID will serve to access INITIALIZE.2 indirectly by executing a connector program which resides in BASIC.DR. The system manager will be responsible for maintaining the con- nector program while INITIALIZE.2 will be maintained as part of the AUTORUN subsystem.

The data acquisition and reduction module for the various ISCN systems will cause the most difficulties in the maintenance and expansion of the system. All ISCN programs in one system will be identical to those in another except for the two programs which are responsible for acquiring and reducing data. These are ISCN.03 which is the instrument checkout program and ISCN.04 which is the IRCL router and major measurement program. All instrument related parameters are defined in ISCN.03 and the checkout session is tailored specifically to the automated instrument. The ISCN.04 program will, however, remain substantially the same from one instrument system to the next with the exception of lines running from 3000 to 4000. This block of code will be tailored to the specific automated instrument.

The following instrument-independent programs will be stored in BASIC.DR and will be used by all ISC systems: ISCN.00, ISCN.01, ISCN.02, ISCN.05, ISCN.06, ISCN.07, ISCN.08, ISCN.09, ISCN.10, ISCN.11 and ISCN.12. The AUTORUN and INITIALIZE.2 programs are the same for all ISCN systems, but they must reside in the particular instrument directory. The ISCN.03 and ISCN.04 programs will be different for each particular instrument application and will be contained in the particular instrument directory.

TABLE 2

AUTOMATION SYSTEM PROGRAMS AND FILES

Name	Type	Description
AUTORUN	Program	Automation system starter - chains to ISCN.00;
INITIALIZE.2	Program	Special access program for parallel/off-line plotting of calibration curves - chains to ISCN.08;
ISCN.00	Program	Automation System Command Level (ASCL) router - chains to ISCN.01, ISCN.03, ISCN.04, ISCN.08, ISCN.09, ISCN.10, and ISCN.11;
ISCN.01	Program	Translate Run File and produce wheel pattern - chains to ISCN.02;
ISCN.02	Program	Control input of pre-RUN parameters - chains to ISCN.03;
ISCN.03	Program	Define instrument parameters and check out instrument - chains to ISCN.00 or ISCN.04;
ISCN.04	Program	Major RUN control program (IRCL) - chains to ISCN.00, ISCN.05, ISCN.06, ISCN.07, ISCN.08, ISCN.09 and ISCN.12;
ISCN.05	Program	Calibrate standards-chains to ISCN.04;
ISCN.06	Program	Generate interim RUN status report - chains to ISCN.04;
ISCN.07	Program	Final Report and/or Run Results File generator (FCL) - chains to ISCN.04;
ISCN.08	Program	Calibration curve plotter - chains to ISCN.00 or ISCN.04;

TABLE 2 (Contd.)

ISCN.09	Program	Raw peak-array plotter (where applicable) - chains to ISCN.00 or ISCN.04;
ISCN.10	Program	Temporary Run File developer - chains to ISCN.00;
ISCN.11	Program	Report and data file dumper - chains to ISCN.00;
ISCN.12	Program	RUN editor - chains to ISCN.04;
ISCN.QC	Data file	Quality Control parameters for the temporary Run File developer;
ISCN.ID	Data file	Sample identifiers for a RUN;
ISCN.DF	Data file	Sample absorbance, volume and concentration data for a RUN;
ISCN.TR	Data file	Transfer file for RUN data shared by multiple programs;
ISCN.PT	Data file	Calibration curve plot file;
ISCN.PK	Data file	Raw peak-array plot file (where applicable);
ISCN.LC	List file	Calibration report;
ISCN.LD	List file	Data dumper report;
ISCN.LE	List file	Run File Error list;
ISCN.LF	List file	Final Report;
ISCN.LL	List file	Line printer list file of raw peak data;
ISCN.LP	List file	Line printer list of test peak data;
ISCN.LR	List file	Interim RUN status report;
ISCN.LW	List file	Wheel pattern report;
-.RF	List file	Run File;
-.RR	List file	Run Results File.

REFERENCES

1. Initial Data Element Dictionary (DED) for the Sample File Control (SFC) System, Auerback Associates, Inc., December 1978.

2. Methods for Chemical Analysis of Water and Wastes, 1979, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio.

3. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, 1979, EPA-600/4-79-019, U. S. Environmental Protection Agency, Cincinnati, Ohio.

4. Rose, Thomas F. The User's Guide for the Sample File Control System on the Data General NOVA 840, July 1980. University of Cincinnati Computing Center, Cincinnati, Ohio.

5. Teuschler, John M., Chris Cusiak and Kevin Geiger. A Computer Interface for a Perkin-Elmer 5000 Atomic Absorption Instrument, January 1980. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio.

APPENDIX A

Purpose and Usage of Calibration Standards, Check Standards, Lab Reagent Blanks and Lab Control Standards as They Relate to Sample File Control

Many methods have been established to determine the concentrations of pollutants in environmental samples. The majority of these methods utilize standard calibration as the basis for computing the concentration of an analyte in an "unknown" sample. The Beer-Lambert Law supplied the mechanism for developing "standard curves" relating known concentrations to measured absorbances. Today, it is known that many types of phenomena produce signals which are directly proportional to the concentration of a particular analyte in an environmental sample.

In this discussion, it will be assumed that for a given analyte, there exists a method which can be used to produce a response or signal which is directly proportional to the concentration of the analyte being measured, such that:

```
c  =  P * s;
where:
c     is the concentration of the analyte;
P     is the constant slope of the line; and
s     is the raw or reduced instrument response to the analyte in
      question.
```

This ideal relationship is seldom achieved in the laboratory because of the non-linearity of instruments used in measuring the phenomena. The chemistry used in producing the phenomena may also contribute to the non-linearity of the system. In general, however, our discussion will assume some form of linearity between s (signal) and c (concentration) even if the linearity is piecewise or linear over a discrete concentration range.

Preparing Standards

A "stock" standard solution is made by adding a known mass of an analyte to a "blank" solution of known volume. The stock solution is usually highly concentrated so that it can be titrated into various volumes of the "blank" solution to form a number of standards having different concentrations. For a given method, these standards are made up with concentrations starting at the "detection limit" of the method and increasing to a maximum allowable concentration for the method. In other words, the standards span the working range dictated by the analysis technique. In addition, the "blank"

solution is also maintained, untainted by analyte, to serve as a zero calibration standard.

Characteristics of the Standard Curve

Figure A-1 shows the relationship between s and c for a number of standards. Note that S_0 (the blank solution) will normally exhibit an instrument response even though the analyte in question has not been added. S_1 represents the lower limit of the method and is called the lowest measurable standard, while the span from S_1 to S_n is called the optimum concentration range or working region for a method.

Computing Concentration by Interpolation

An analyst will prepare enough standards to adequately represent the working region for a particular method. This may involve grouping some standards where obvious non-linearity is present.

By measuring an adequate number of standards at appropriate intervals, an analyst can then proceed to measure unknown samples and perform an interpolation between the bracketing standards in order to obtain the concentration of the unknown. Figure A-2 depicts the interpolation algorithm.

As long as standards are accurately produced and enough of them are measured to represent method non-linearity, then the use of interpolation is a desirable technique for computing unknown sample concentrations.

Calibration by Statistical Regression

More sophisticated techniques are available which fit the standard points to a polynomial equation of the form:

$$c = P_0 + P_1 * s + P_2 * s^2 + P_3 * s^3 \ldots\ldots$$

Regression usually involves complex matrix manipulations and normally requires a computer to perform the operation. The least squares regression technique remains powerful only when linearity is essentially maintained. The technique does not adequately represent most response phenomena over large concentration intervals unless the response is linear.

There are some advantages to using regression to calibrate the standards. For example, a by-product of the regression algorithm is a number which represents the fitting error of the calibration equation. The statistical term for this value is the relative standard error of estimate, and the value may be expressed in percent. If the fitting error is high, then the analyst may want to review the degree of fit being used. A large fitting error may also be the result of a poorly produced or erroneously measured standard.

Another advantage of using regression to calibrate standards is that the technique tends to smooth out precision errors and preparation and measurement variability.

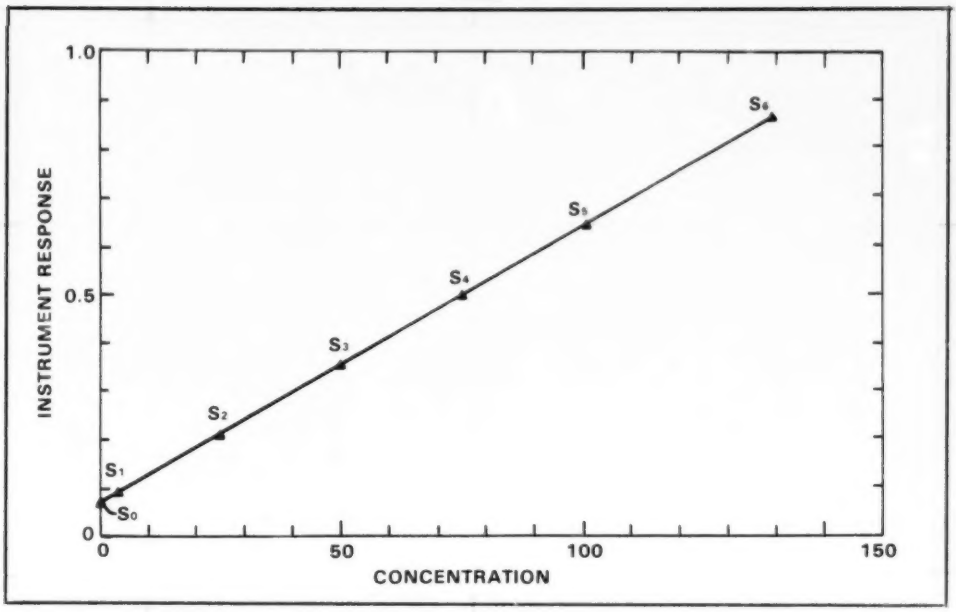


Figure A-1. Characteristic standard calibration curve.

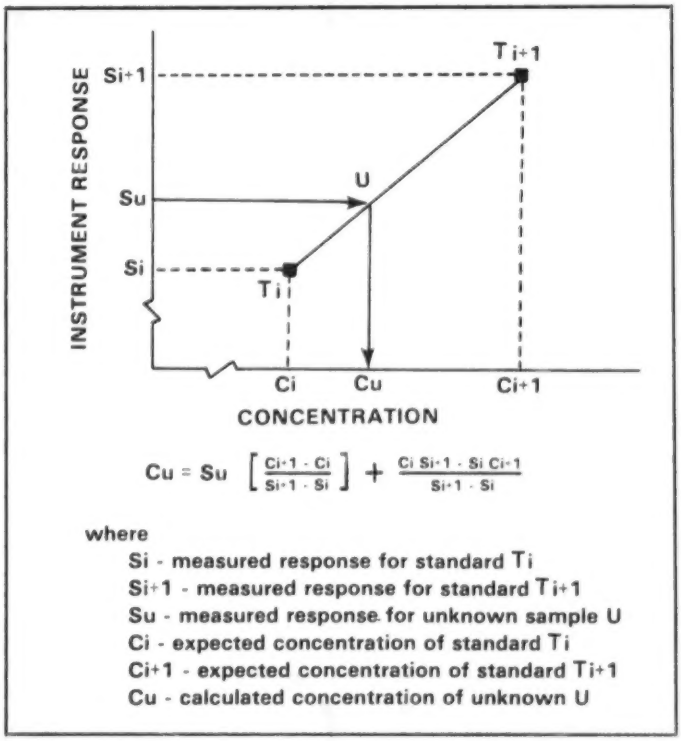


Figure A-2. Concentration computed by interpolation.

Least squares regression is a useful tool for relating concentration to signal, but it can easily be misused. For example, any three points can be fit exactly (fitting error =0%) with a second degree (quadratic) equation. More measurements are required before an analyst can be sure of the shape of the curve and the acceptability of the prepared standards. Current recommendations regarding the fewest number of standards needed to utilize regression are stated below in terms of the degree of equation required:

1st degree - S_0, S_1, S_2, S_3
2nd degree - S_0, S_1, ... S_4
3rd degree - S_0, S_1, ... S_5

Notice the zero standard is required in all cases and that none of the computational techniques thus described requires curve intersection with the origin.

Check Standards as a Measure of Run Stability

A "run" is defined as an analytical sequence within which various types of samples are measured for the purpose of evaluating precise and accurate concentrations of a particular analyte in a batch or group of environmental samples. A run may take an hour or a week depending on the time required to process a single sample. A run is usually composed of a set of calibration standards, unknown samples from various sources, and quality control (QC) samples which are used to evaluate the precision and accuracy of the reported unknown concentrations. Normally, standards are measured at the beginning of the run, and a standard curve or equation is produced. Unknown and QC samples are then measured and concentrations are calculated based on the chosen computational mode. During the course of the run, an analyst will usually measure one of the midrange standards and compare the signal with that obtained from the first measurement of the standard. This comparison is often expressed as a "percent recovery of the instrument check-standard." A poor recovery for a check standard measurement indicates system instability and in some cases may warrant the termination of the run or a recalibration of the standards.

Some analysts use check standard measurements to modify their standard curves during the run. There are two problems with this procedure. First, There is no mathematically viable technique for adjusting a multipoint calibration curve through the remeasurement of one of the points, without reverting to a single point calibration system. Second, the poor check standard recovery may be due to a combination of factors which are not necessarily predictable over time.

Let it be stated here, as a precept, that check standards will only be used to determine when it is necessary to prepare a new standards curve. The analyst will be responsible for defining and maintaining limits, within which, check standard recoveries must fall during the course of a stable run. Any excessive variability of the check standard recovery will warrant run termination and recalibration of the standards.

Lab Reagent Blanks and Lab Control Standards

A lab reagent blank or LRB is defined as a "blank" solution which as been treated exactly as a lab sample for the parameter being measured, including all preanalysis processing. The LRB is the reported concentration of a reagent blank. The measurement of the reagent blank is used as an indication of contamination introduced in the sample preparation procedures. If the reagent blank measurement is high, then any samples which were similarly processed may also exhibit higher measurements due to the same contamination. This may be checked by comparing the reagent blank signal with that obtained from the measurement of the zero-calibration standard. Detectable differences at a low level, however, do not warrant the correction of other sample results without further evidence.

A secondary proof for contamination is required. This is normally accomplished by measuring a control standard. A control standard is defined as a calibration standard which has been carried through the same sample preanalysis processing in the laboratory that is used for unknown samples. The control standard is similar to the reagent blank in that both are subject to approximately the same contamination due to prerun preparation. The measured concentration of the control standard (LCM) is usually compared with its theoretical or expected concentration (LCT). The percent recovery of LCM with respect to the expected concentration (LCT) should be compared to past control standard recoveries in order to ensure quality control. At the same time, a comparison may be made between the signal obtained for the control standard and that obtained from its original calibration standard. If the control standard signal is higher than the standard from which it emanates, then it is possible that some prerun contamination has been introduced. If the reagent blank also measures higher than the zero-standard, then there is good reason to believe that a systematic error has been introduced due to preparation. In this case, it is possible to utilize the reagent blank signal to adjust signals obtained from unknown samples so that the effect of the contamination is eliminated. The proposed procedure for performing this adjustment is to compute the difference between the reagent blank signal and the zero-standard signal and subtract this difference from unknown and QC sample signals. The concentrations of the unknowns may then be recalculated with the chosen computational mode (e.g. interpolation or regression).

The fact that an analyst has adjusted sample signals because of high reagent blank signals will be invisible to the Sample File Control system. However, it is most important that LCM and LRB concentrations be reported exactly as computed without adjustment. The Sample File Control System will accumulate statistics for these QC items and update the control limits for them.

The in-run value of reagent blanks and control standards has been discussed while skirting the issue of validating QC results through externally computed limits. Preparation or method error is not the only problem detected by the measure- ment of reagent blanks and control standards. Due to the nature of the calibration curve or equation, other

errors arise due to calibration standard preparation or measurement. A control standard may be measured and its recovery may be checked against expected limits in order to validate the standard itself as well as the curve or equation being used in the computa- tion.

A more subtle problem of curve fitting error exists when regression is used to fit the standards. This problem usually causes erroneous results for low level samples. It is easily recognizable if a reagent blank is measured and the computed concentration is compared with the control limits for the LRB.

If the reagent blank concentration or the control standard recovery is unexplainably out of control with respect to historically defined limits, then the run should probably be terminated.

APPENDIX B

Proposed Sample Dilution Procedures and Sample Spiking Procedures for Preparing Samples and Reporting Concentrations to Sample File Control

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Normal Sample Dilution Procedures

In the normal standard calibration procedure, it is conceivable that the measured concentration of an unknown sample may exceed the limits of the standards curve. For example, if a sample produces an absorbance measurement significantly higher than that obtained from the highest standard in the calibration set, the analyst is usually required to dilute the sample and remeasure it. This dilution should effectively place the unknown within the standards range, and a valid concentration may then be measured. Once this concentration is obtained, it must be adjusted by the dilution factor so that the reported concentration will relate to the original unknown and not the diluted unknown. Figure B-1 details the procedure.

Duplicate Sample Dilution Procedures

A complicating factor, regarding dilutions, arises when the sample is a member of a duplicate quality control set. If the duplicate samples measure off-scale, all existing measurements on the samples must be discarded. The analyst will then be required to dilute and remeasure each member in the duplicate set. This is normally a simple procedure and is detailed in Figure B-2.

Normal Sample Spiking Procedures

Before discussing dilution procedures for spiked samples, the normal procedures for dealing with spiked sample set up and measurement will be described.

In general, a spiked sample quality control set is formed by taking equal volumes of an unknown sample and spiking one aliquot with a specific volume of a spiking solution whose concentration is known. The volume of the original unknown and that of the spiking solution have a direct affect on the values reported for the spiked sample quality control set. Figure B-3 depicts the normal spiking procedure.

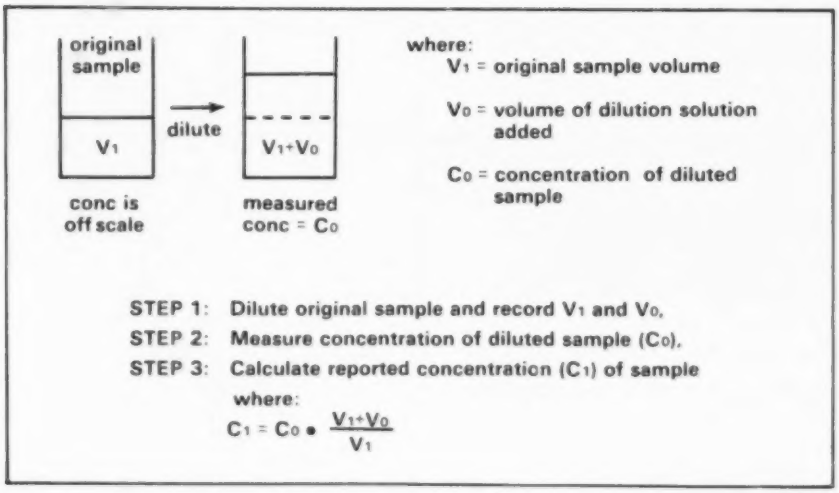

Figure B-1. Normal dilution procedure.

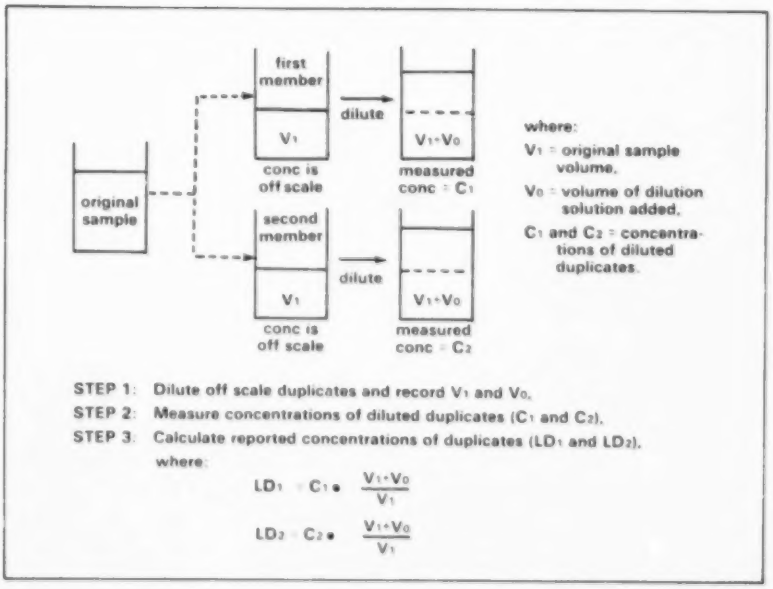

Figure B-2. Duplicate dilution procedure.

The following definitions specify the quantities which are reported as a result of the spiking procedure:

LSO is the measured concentration of the original unknown,
LSF is the measured concentration of the spiked sample,
LSA is the amount by which the concentration of the original sample will increase due to the addition of the spike.

In terms of the quantities defined in Figure B-3, the following equations detail the reported values for LSO, LSF, and LSA:

1. $LSO = C_1$

2. $LSF = C_3$

3. $$LSA = \frac{C_2 . V_2 - C_1 . V_2}{V_1 + V_2}$$

In the case where a solid or minute volume of a highly concentrated solution is added as the spike, the mass of the added analyte (M_1) replaces the quantity $C_2 . V_2$ giving the following reported result for LSO, LSF, and LSA:

4. $LSO = C_1$

5. $LSF = C_3$

6. $$LSA = \frac{M_1}{V_1}$$, where $V_2 = 0$

The % Recovery of the spike is defined as follows:

7. $$\% \text{ Rec. of spike} = \frac{LSF - LSO}{LSA} \times 100$$

The following procedures will be followed in order to produce reported results for LSO, LSF, LSA, and % Rec. of spike:

STEP 1: Spike sample aliquot as shown in Figure B-3

STEP 2: Record V_1, V_2, and C_2, or if V_2 is negligible, record M_1 and V_1

STEP 3: Measure concentrations C_1 and C_3

STEP 4: Compute the reported values for LSO, LSF, and LSA using equations 1, 2, and 3; if the volume V_2 is assumed to be zero, equations 4, 5, and 6 may be used

STEP 5: Compute the percent recovery of the spike using equation 7.

Procedures for Spiking Diluted Samples

If either measurement of the spiked sample set exceeds the highest calibration standard, the spiked sample set must be reformulated and the existing data on the sample must be discarded. Figure B-4 shows one acceptable technique for reformulating the spiked sample set through dilution.

The following procedures should be followed in order to produce reported results for LSO, LSF, LSA, and percent recovery of the spike under the conditions of dilution shown in figure B-4:

STEP 1: Dilute the sample aliquots with the dilution solution.

STEP 2: Spike one diluted aliquot as shown in Figure B-4.

STEP 3: Record V_0, V_1, V_2, and C_2, or if V_2 is negligible, record M_1, V_1, and V_0.

STEP 4: Measure concentrations C_1 and C_3.

STEP 5: Compute the reported values for LSO, LSA, and LSF using the following equations:

8. $$LSO = C_1 \cdot \frac{V_1 + V_0}{V_1}$$

9. $$LSF = C_3 \cdot \frac{V_1 + V_0 + V_2}{V_1 + V_2}, \text{ or } C_3 \cdot \frac{V_1 + V_0}{V_1} \text{ if } V_2 = 0$$

10. $$LSA = \frac{C_2 \cdot V_2 \cdot V_1 - C_1 \cdot V_1 \cdot V_2 - C_1 \cdot V_2 \cdot V_0}{V_1 \cdot V_1 + V_1 \cdot V_2}, \text{ or } \frac{M_1}{V_1} \text{ if } V_2 = 0$$

STEP 6: Compute the percent recovery of the spike using equation 7 mentioned above.

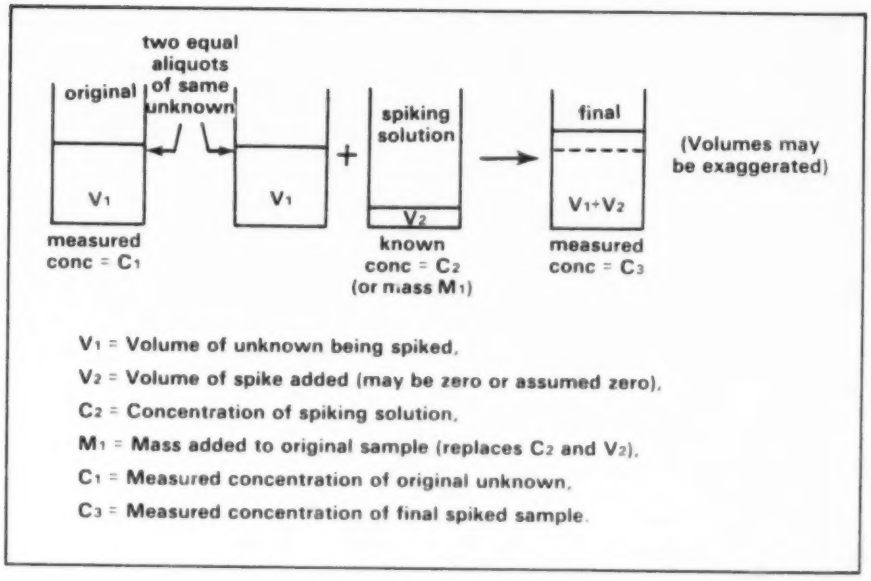

Figure B-3. Normal sample spiking.

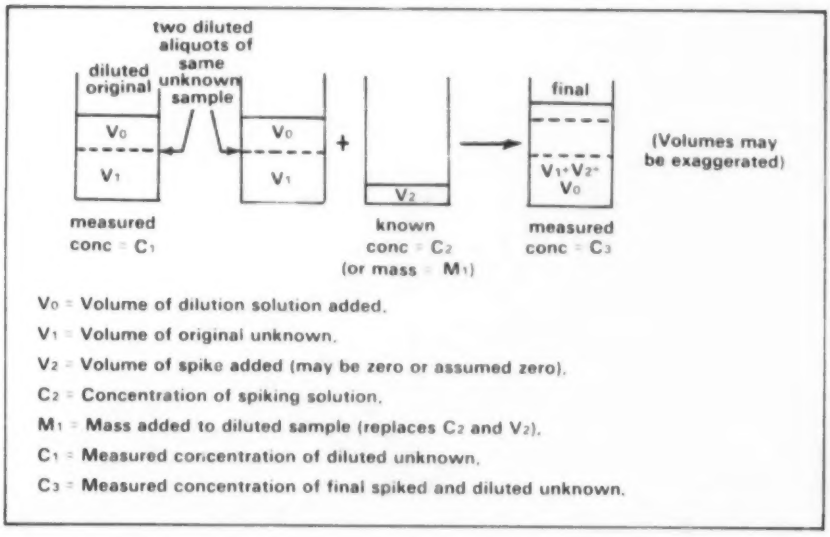

Figure B-4. Spiking a diluted sample.

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